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ANALYSIS OF WATERS OF THE YELLOWSTONE NATIONAL PARK.

WITH AN ACCOUNT OF THE METHODS OF ANALYSIS EMPLOYED.*

BY F. A. GOOCH AND J. E. WHITFIELD.

INTRODUCTION.

The series of waters—forty-three in all—of which it is the purpose of this paper to give some account, comprises typical representatives of the geysers, hot springs, mud springs, cold springs, and surface waters found within the limits of the Yellowstone Park. It embraces collections made during the field seasons from 1883 to 1886 under the direction of Mr. Arnold Hague, geologist in charge of the Yellowstone Park Division, and submitted to us for analysis. In our account we have to deal simply with the results of analysis and with the analytical processes we have used—with the latter on the ground that some knowledge of the methods employed in the examination and of the quantities of material at our disposal is essential to a correct understanding of the meaning and value of the analyses themselves. Discussions of the broader chemical aspects of the problems which are involved in a thorough consideration of the relations of these waters, of correlative matters of geological interest, and of the physical phenomena belong properly to others.

The difficulties in the way of collecting and transporting large quantities of water in a region comparatively inaccessible were such that the number of large samples, for the more complete analyses, was restricted to a half-dozen ; but, though at the beginning of the examination it was supposed that a partial

*From the Bulletin of the U. S. Geol. Survey, No. 47.

analysis, such as might be made with smaller amounts of material, must suffice, it happened, fortunately, that the development of certain methods as the work progressed has so far extended the reach of the analyses of the smaller samples that from them it is possible to deduce results of fair accuracy, and, if such elements be left out of consideration as are shown by the more elaborate analysis to be present either not at all or only as traces in closely comparable waters, nearly as comprehensive results as those drawn from the examination of the larger samples.

OPERATIONS IN THE FIELD.

The examination of the waters was necessarily confined in the field to a very few points. Note was made of the *clearness* or *turbidity*, the *reaction* to litmus and turmeric, the *temperature*, and the presence or absence of *hydrogen sulphide*. The tests for the last were the sense of smell and the action of the water upon a polished silver blade.

The operations of collecting and preparing the samples for transportation were carried out as simply as possible. The water was bottled in boxed demijohns when it was possible, and otherwise in beer bottles. In every case great care was taken to clean the receptacle thoroughly and rinse before bottling with some of the water to be examined. In the greater number of cases the waters were so marvelously clear that filtration was entirely superfluous. When, however, the need and opportunity occurred the waters were filtered previous to bottling. All samples were corked tightly and sealed hermetically.

The size of the sample varied from a half liter to one hundred liters, according to the purpose for which it was taken, but the great bulk of the largest samples was in every case excepting one reduced greatly by the evaporation of the main portion of the sample previous to bottling. In the one exceptional case the sample was taken for the specific purpose of examining it as to its contents in the heavy metals, and was kept for subsequent reduction in porcelain in the laboratory, in order that the danger of contamination by the tin or copper of the tin lined still which was employed to effect the reduction in the field might be precluded. No attempt was made to fix with more than a rough approximation the quantities of water evaporated. The concen

trated waters, with the rinsings of the still in the natural water of the charge (such portions of the residue as adhered to the walls of the vessel having been dislodged as completely as possible by gentle friction with a rubber or the ball of the finger), were carefully bottled and sealed, and the determination of the degree of concentration was left to be effected in the laboratory in a manner to be described later. A portion of the water represented by the concentrated sample was always reserved in the natural condition for examination.

For the determination of hydrogen sulphide special samples of from 600 cm^3. to 700 cm^3. were generally taken, and to each, if the water did not carry free sulphur, was added before corking and sealing a small amount of lead acetate in crystals. Doubtless it might have been well to fix the hydrogen sulphide similarly in the case of waters carrying free sulphur, having first separated this by filtration; but on the few occasions when that need arose the means of removing the sulphur were not at hand. In these cases, therefore, the samples were simply bottled and sealed with care, and kept for examination in the laboratory.

Nearly all the waters examined were hot when the samples were taken and not surcharged with carbonic acid. It was considered unnecessary, therefore, under the circumstances, to take a special sample for the determination of the carbonic acid and treat it in the field so as to fix that gas in insoluble form, but for this estimation reliance was placed upon the general sample which, as has been said, was corked and sealed with great care.

OPERATIONS IN THE LABORATORY.

The operations in the laboratory comprised the determination of the specific gravity of the waters and, in the concentrated samples at hand, the estimation of the following substances:

* Silicic acid.	Iodine.	Barium.
Titanic acid.	Fluorine.	Strontium.
* Sulphuric acid.	* Sulphur.	* Calcium.
Sulphurous acid.	* Hydrogen sulphide.	* Magnesium.
* Carbonic acid.	Copper.	Cæsium.
Nitric acid.	Lead.	Rubidium.
Nitrous acid.	* Arsenic.	* Potassium.
Phosphoric acid.	Antimony.	* Sodium.
* Boric acid.	* Iron.	* Lithium.
* Chlorine.	* Aluminum.	* Ammonia.
Bromine.	Manganese.	* Albuminoid ammonia.

In analyses for which the natural water alone was available those substances only were sought which are marked in the table with an asterisk (*).

The determination of what is known as the *total residue*, the amount of solid matter left upon evaporating a known amount of water, is usually regarded as desirable, if for no other reason than to serve as a control of the summation of the determination of the individual constituents; but the complexity of these waters and the relations of the combined salts are such that it is not possible to arrive at the same end in two successive treatments by evaporation and desiccation of the residue. The action of free silica, which is an abundant constituent of the waters, is to set free during the process carbonic acid from the carbonates and boric acid from the borates; to magnify the tendency of the chlorides of calcium, magnesium, and lithium to exchange chlorine for oxygen, and, if the temperatures be pushed sufficently high, to dehydrate the silica and to volatilize sulphuric acid. Furthermore the extent of such action is variable and indeterminate. There is obvious reason, therefore, for the difficulty which we experience in obtaining residues of constant composition; and similar obstacles, as we found practically, oppose the attempt to reduce the residue to a definite condition by evaporation and ignition with sulphuric acid in excess. By means of no device which occured to us (and several lines of experimentation which need not be mentioned here were followed) were we able to apply, in any form, this general method of control. The proof of the accuracy of our analytical work rests, therefore, upon the agreement of duplicate determinations.

For convenience in the matter of arrangement, we describe first those methods of analysis which were applied to waters in the natural condition; and the account of the treatment of concentrated waters follows. Methods for the determination of certain substances are described under both heads. For when the entire work was done upon the natural water the mode of examination was sometimes made, of necessity, by processes different from those which were applied in the examination of concentrated water; and under reversed conditions the converse was true. In some of the earlier analyses methods were employed which later

experience taught us might be advantageously replaced by processes which were developed subsequently. In the account of the methods employed all of these are placed under the proper head; and in the statement of analyses mention is made, when there might be doubt of the particular method adopted.

TREATMENT OF NATURAL WATERS.

Specific gravity.—The figures which stand for the specific gravity of a water represent the weight of any volume of the water under consideration at 15.5° C. compared with the weight of the same volume of distilled water at the same temperature. The determinations were nearly all made with a Geissler bottle, fitted with a ground tubular stopper and cap, and a thermometer. In a few of the earlier determinations a bulb-apparatus similar in general principle, but wanting the immersed thermometer, was employed. The temperature of the water in the bottle was taken immediately after the final adjustment of volume, and the latter was effected instantly and without handling after the filled bottle had been permitted to stand a few moments to bring about an equal distribution of temperature. When the bulb-apparatus was employed the temperature of the water was taken before filling the bulb and after weighing, and the mean of the two readings was assumed to be the temperature of the water at the time when the volume was adjusted. The weight shown by the balance was taken as the weight of the water which would fill the apparatus at the temperature noted. The weight of the same volume of the water at 15.5° C. was calculated from the weight observed, upon the assumption that the co-efficient of expansion of the water under examination was the same as that of pure water. The rates of the value thus found to the value found by a similar treatment of pure water is the specific gravity at 15.5° C. It is obvious that, in order that the assumption of the identity of the co-efficients of expansion of the water under examination and of pure water may be the occasion of the least error, the observed temperature must be as near 15.5° C. as may be; and that the temperature of the balance-room should be not far from the same, in order that errors which may arise from changes of temperature in the water during the manipulation, and from the establishment of air-currents in the balance-case may be less liable to occur. So

far as we were able these sources of inaccuracy were guarded against, but the conditions under which the work was performed necessitated the application of corrections rather greater than we should wish. The figures which represent the specific gravity in the statement of the analyses are probably not accurate beyond the fourth decimal place.

Hydrogen sulphide and free sulphur.—Hydrogen sulphide in waters which did not carry free sulphur was determined in the special sample to which lead acetate had been added in crystals at the time of bottling. Before breaking the seal of the bottle a scratch was made upon the neck on the line of the meniscus of the water within, and the volume of the sample—usually about 600 cm^3—was determined subsequently with sufficient accuracy (the amount of hydrogen sulphide present being always trifling) by finding the volume of water necessary to fill the bottle to the mark. The precipitate which had been formed in the bottle by the addition of lead acetate was left undisturbed at first, and the supernatant liquid decanted as completely as possible. Then the precipitate was easily removed, and for the most part dissolved by means of a little sodium hydrate in solution, and the solution and remaining precipitate were added to the water previously decanted. At this point the lead sulphide showed itself, if any was present, and was collected without difficulty by filtering the alkaline liquid in which it floated through a felt of asbestos in a perforated crucible, in the manner described by one of us previously.[1] The felt and the adherent precipitate were transferred to a beaker, treated with potassium chlorate and hydrochloric acid to oxidize the sulphide, and the sulphuric acid thus formed was determined by precipitation as barium sulphate. From waters carrying free sulphur that element was separated by filtration. The residue, after washing sufficiently to remove the sulphates, was treated with hydrochloric acid and potassium chlorate, and the resulting sulphuric acid was determined as barium sulphate. The filtrate from the sulphur contained the hydrogen sulphide, if it was present in the sample, and this was removed by lead acetate and estimated in the manner described above.

Sulphurous acid.—The test for sulphurous acid was made in

[1] Am. Chem. Jour., vol. 1, 1879, p. 317.

the case of a few waters which did not carry more than a trace of hydric sulphide by titrating 200 cm^3 of the water, acidified with sulphuric acid, with a standardized solution of potassium permanganate. In none of these was a reducing action found greater than was attributable to the action of the hydrogen sulphide and arsenic (assumed to be in the condition of arsenious acid) known to be present.

Sulphuric acid.—The sulphuric acid was precipitated from a known weight of water, after acidifying distinctly with hydrochloric acid and heating to boiling by means of barium chloride. After cooling and settling, usually after twelve hours, the supernatant liquid was decanted as completely as possible from the precipitate and passed through an asbestos felt in a perforated crucible. The precipitate remaining in the beaker was boiled with a small portion of strong hydrochloric acid, and the latter was removed by evaporation on the water-bath. Treatment with hot acidulated water left the precipitate as pure as barium sulphate may be, and ready for collection upon the same felt through which the supernatant liquid had been poured. Thorough washing, ignition, and weighing completed the operation.

Nitric and nitrous acids.—In examining the waters as regards their contents in nitric and in nitrous acids a modification of the indigo method was followed. The action of these acids upon indigo is known not only to be variable with the conditions of dilution, temperature, and the proportion of sulphuric acid added, but also to be dependent upon the order in which the solution of indigo, the sulphuric acid, and the nitrous or nitric acid are intermixed. On this account great care was taken to secure uniformity of treatment of the standard solutions and the water under the test. For preliminary testing a portion of water was reduced by evaporating to a volume of 50 cm^3. cooled to the temperature of the air, introduced into an Erlenmeyer flask, and mixed with an equal volume of strong sulphuric acid. To the mixture the solution of indigo was added until a distinct tinge of green became visible. In this manner the amount of the solution of indigo from which a given amount of the water under test would discharge the color became known approximately. For the exact test the portion of water, after evaporation and cooling, was

brought to a volume which was less than 50 cm^3. by an amount equivalent to the volume of the solution of indigo used in the preliminary test. To the portion thus prepared were added 50 cm^3. of strong sulphuric acid at once, and the solution of indigo as rapidly as the catching of the exact point at which the tinge of green appeared would permit. At the instant of coloration the flask with its contents was placed in a bath of paraffine heated to 140° C. The volume of the solution of indigo which had been added was noted and, if at the end of five minutes' heating in the bath the tinge of color was still visible, this volume was taken as the measure of the nitrates and nitrites in the water. If, on the other hand, the color vanished during the heating, it was reproduced by the addition of more indigo, and the digestion in the bath at 140° C. was prolonged during another interval of five minutes.

This mode of treatment was repeated until the coloration was permanent during the interval of digestion. The total volume of the solution of indigo employed was recorded, and its value in terms of nitric acid determined in the manner to be described.

As Warrington has shown,[1] the oxidizing power of nitric acid upon indigo decreases as the dilution of the solution in which the action takes place increases. It is plain, therefore, that in order that the exact value of the volume of the indigo solution employed in the test may be known, the amount of nitric acid needed to discharge the color from an equal amount of the same solution under the conditions of the test must be determined. To effect this determination we made use of a solution of potassium nitrate of known strength, and found by experiment the amount of it which, mixed with 50 cm^3. of strong sulphuric acid, with water enough to bring the final volume to 100 cm^3., and with the amount of the solution of indigo employed in the test, was just adequate to discharge all color excepting the faint characteristic tinge which must remain unchanged after five minutes' heating. The oxidizing power of the nitrates and nitrites in the water is exactly measured by the amount of nitric acid in the quantity of the standard nitrate solution which is required to produce the end-reaction. The solution of potassium nitrate which we used

[1] J. Chem. Soc. London, 1879, p. 578.

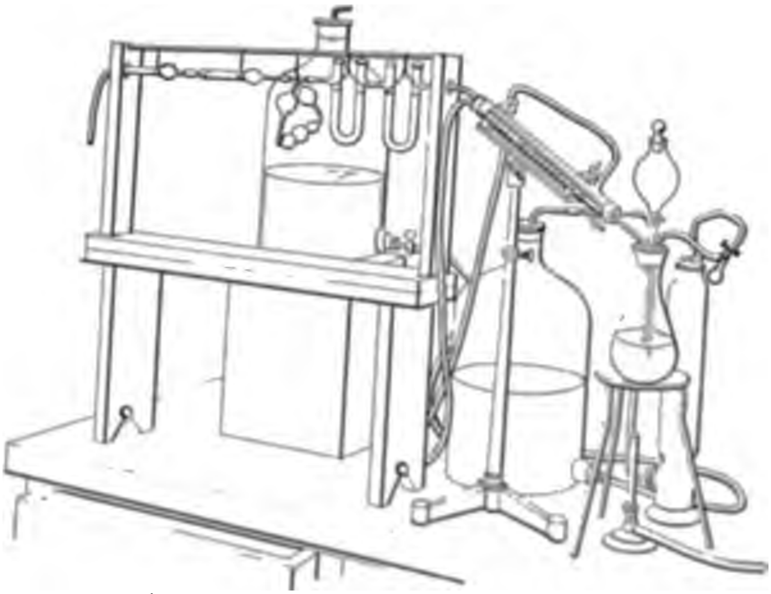


Fig. 1.—Apparatus for the Estimation of Carbonic Acid

Fig. 2.—Apparatus for the Estimation of Boric Acid

contained 0.0001 g. to 1 cm^3. The mode of manipulation which we adopted keeps the proportion of strong sulphuric acid added to the mixture (excepting the preliminary tests) always the same, and consequently the heat generated in the reaction is the same always. The order in which the reagents are brought together is the same in all cases, and sources of inaccuracy due to the variability of the action of nitric acid with the degree of dilution are eliminated.

Carbonic acid.—The greater number of the waters with which we have had to deal were hot when the sample was taken, and not surcharged with carbonic acid. In view of the circumstances under which the waters were collected it was thought that a portion of the general sample, which was carefully sealed, might be made to serve the purpose of determining the carbonic acid contained in the water, instead of portions taken specially for that end and treated in the field with a mixture of ammonia and calcium chloride to fix the carbonic acid gas. Accordingly dependence was placed upon the large general sample for this estimation. The presence of free silica in the waters and the complexity of composition place so great an obstacle in the way of determining the *free carbonic acid* that the attempt to make the estimation was not undertaken. The method which we adopted for the determination of the *total carbonic acid* is that well-known process which rests upon the boiling of the acidified liquid and the absorption and weighing of the gas evolved. The apparatus which we employed, differing somewhat from that in ordinary use for this purpose, is shown in Fig. 1. It consisted essentially of a flask in which to boil the water, a cooler to return the condensed steam to the flask, a U tube filled with anhydrous cupric sulphate to intercept traces of hydrochloric acid which might escape condensation in the cooler, a second U tube charged with calcium chloride to dry the gas, potash bulbs to retain the carbonic acid, and two tubes filled with calcium chloride, one of which was to retain moisture carried over from the potash bulbs and was weighed with them, the other serving to protect the weighed tube from the action of the external air. Through the rubber stopper of the flask passed the tube of a separating funnel, the end of the condensing tube, and a tube which connected the

interior of the flask with the system of bottles and scrubbing tower, which furnished at will and under slight pressure a supply of dry air free from carbonic acid.

Before beginning the operation of determining the carbonic acid in a water the atmospheric air was expelled from the apparatus and replaced by pure air free from carbonic acid. The stop-cock in the funnel tube and the connection with the aspirator having been closed, the water started through the cooler, and the joint broken between the potash bulbs and the drying tube immediately preceding it, a few cubic centimeters of distilled water were put into the flask and boiled. The steam generated drove the air from the flask, and that remaining in the tubes was swept forward by pure air admitted from the aspirator. The outer opening of the tube at the end of the line was closed, the source of heat removed, and the flask allowed to fill with air from the aspirator. The whole apparatus having been filled thus with air free from carbonic acid, the water to be examined—we found 200 g. to be a convenient amount—was introduced into the flask through the funnel tube and followed by a little boiled water and a sufficiency of sulphuric acid, care being taken to exclude the outer air during the operation. The connection between the line of drying tubes and the absorption bulbs was re-established, the liquid in the flask was brought to the boiling point, and, to secure constant and quiet ebullition as well as to aid in transferring the carbonic acid from the flask to the absorption bulbs, a current of pure air was sent slowly through the boiling liquid which covered the end of the aspirating tube and that of the funnel tube. At the end of half an hour the burner was removed from under the flask and the apparatus was permitted to cool during another half hour, the current of air being kept in motion through it. The difference between the weights of the potash bulbs, with the drying tube which accompanies them, before and after the operation, was taken to be the weight of carbonic acid absorbed, and the *total carbonic acid* of the portion of water examined.

When the water under examination contained hydrogen sulphide, a little cupric sulphate was introduced before acidifying into the portion which was to serve for the determination of carbonic acid.

Arsenious acid and boric acid.—To the portion taken for analysis—usually 500 g.—hydrochloric acid was added in distinct excess, the temperature of the liquid was raised to 70° C., and the source of heat having been removed, hydrogen sulphide gas was introduced in a gentle current for several hours. The precipitation of the arsenious sulphide began at once, but the action of the precipitant was continued to insure completeness. The precipitate was collected upon an asbestos felt, washed, and dissolved in fuming nitric acid. The solution in nitric acid was evaporated to dryness, the residue was dissolved in hydrochloric acid, and from this solution, made strongly ammoniacal but kept small in volume, the arsenic was precipitated by the addition of the magnesium chloride mixture and brisk agitation. The precipitated ammonium-magnesium arsenate was allowed twelve hours in which to settle and was then transferred by means of the mother liquid, without the use of other liquid, to a perforated crucible carrying an asbestos felt, washed thoroughly with the mixture of one part of ammonia to three of water, usually employed for this purpose, but with the sparing use of the washing material which this mode of filtration permits, dried carefully, ignited gently at first, and at a low red heat afterward, in an atmosphere of oxygen. The great faults of this process, originally Levol's, have been the solubility of the precipitate in the mother liquor and, more especially, in the ammoniacal wash water, and the difficulty in drying or igniting the precipitate collected on paper. These sources of inaccuracy vanish, however, so completely with the use of the asbestos filter and due care in keeping the volume of mother liquor and the washings small, that the filtrate, freed from ammonia, acidified and charged with hydrogen sulphide, fails to yield arsenious sulphide. As a test for the presence of arsenic the method has enjoyed an excellent reputation, and in the form in which we have made use of it we believe it to be exact.

The filtrate from the precipitate of arsenious sulphide, containing all the boric acid, was nearly freed from hydrogen sulphide by the transmission of air, and the chlorine, with residual traces of hydrogen sulphide, was removed by treatment with silver nitrate and filtration. The filtrate from the precipitated

sulphide and chloride was treated with sodium hydrate, in quantity a little more than sufficient to precipitate the excess of silver, as completely as may be. The whole was evaporated until the precipitate took on a compact and granular form, then filtered, and the filtrate was evaporated nearly to dryness. The residue was treated with nitric acid, first just to acidity, and then with a sufficient (measured) amount to make it certain that the boric acid was entirely free. The concentrated solution of the nitrates, nitric acid, and boric acid was drenched with methyl alcohol to precipitate the first and dissolve the last. The solution of boric acid was quickly separated from the precipitate by means of the vacuum filter, and the residue washed sufficiently with methyl alcohol. When much boric acid is present the precipitated nitrates should be dissolved in water and the process of precipitation with methyl alcohol repeated; but when small quantities are dealt with, as was the case in the most of our work upon these waters, a single treatment leaves no boric acid in the residue. From the solution in methyl alcohol, the boric acid was separated and its amount estimated by a method devised by one of us and described elsewhere in full.[1]

This process consists essentially in distilling to dryness the solution in methyl alcohol, containing free nitric acid and nitrates, and treating the residue by successive drenchings with methyl alcohol and distillations. The distillate, carefully protected, contains at the end of the operation all the boric acid, and this is fixed as calcium borate by the evaporation of the distillate over lime. The difference between the weight of ignited lime and the weight of the same after having been submitted to the action of the alcoholic solution and ignition is the weight of boric anhydride in the solution.

The distillation was effected by means of an apparatus which consists of retort, condenser and bath for heating (Fig. 2). For the last a bath of paraffine is on the whole most convenient. The condenser is set vertically to facilitate changing the level of the retort within the bath, and to secure at the same time continued and thorough washing of the tube by its own condensations. The retort, somewhat like the well known drying tube of

[1] [illegible] Am. Chem. [illegible]
This Journal [illegible]

Liebig in general shape, is easily made of a pipette by bending the tube at one end to a right angle, at the other to a goose-neck, as shown. To the former end is fitted, by a rubber stopper or section of tubing, a glass funnel-tube provided with a stop-cock; the end of the goose-neck passes tightly through a rubber stopper in the upper end of the condensing tube. This is essentially the apparatus; but it is convenient to attach, to receive the distillate, a small Erlenmeyer flask, which moves with the condenser and is joined to it, in the manner indicated in the figure, by means of a thistle tube and a rubber stopper grooved to permit the free passage of air. In carrying out the distillation the liquid is introduced into the retort either by the funnel tube or previous to its insertion, the glass stop-cock is closed, the water started through the condenser, and the retort lowered into the hot paraffine, care being taken to begin the operation with the retort not more than half full and so inclined that only the rear dips below the surface of the bath. If the precaution to heat the retort at the start in this manner be overlooked it may sometimes happen that the sudden and violent expulsion of air through the liquid will carry portions of it bodily into the goose-neck and even into the condenser. With this point considered, the remainder of the operation presents no difficulty and requires little care. The size of the retort may be suited, of course, to the particular case in hand, but for most purposes a pipette of 200 cm^3. capacity makes a retort of convenient dimensions, neither too large for the distillation of small charges nor too small to permit the treatment of 100 cm^3. of liquid comfortably. The tube of the goose-neck should be wide enough to prevent the formation of bubbles in it; 0.7 cm. is a good measure for the interior diameter. It is of advantage to heat the bath to a point considerably above the temperature at which the liquid to be distilled boils—something between 130° C. and 140° C. does very well for water, and is not too high for methyl alcohol—and under such circumstances, and when the retort is entirely submerged, it often happens that evaporation takes place with extreme rapidity from the surface of the liquid in perfect quiet and without actual boiling.

Experiment showed that six successive treatments by drenching with 10 cm^3. of methyl alcohol and distillation to dryness were

more than adequate to remove 0.2 g. of boric anhydride entirely from a residue containing 2 g. of sodium nitrate; but, in order to break up the crusted residue, which by its insolubility in the alcohol might effect to some extent the protection of the boric acid from the action of the alcohol, it was found best to introduce and evaporate 2 cm^3. of water between the second and third, and again between the fourth and fifth distillations. In most of the cases with which we were concerned in the work upon these waters four treatments by drenching and distilling, with one intermediate moistening with water, were entirely sufficient to volatilize the boric acid; but in every case the residue was examined for boric acid by the exceedingly delicate turmeric test, taking care to first oxidize nitrites by means of bromine, and to expel the last before applying the test.

When the solution of boric acid in methyl alcohol is put over lime and evaporated at once some volatilization of the boric acid is apt to take place; but a short period of digestion—from five to fifteen minutes—is sufficient to fix the boric acid completely.

To secure the proper exposure before evaporation, with the least delay, the lime which is to retain the boric acid, first ignited in the crucible in which the evaporation is to be made subsequently, is transferred to the receiving flask attached to the condenser before the distillation is begun, and so has opportunity to exert its action upon the boric acid as the distillation progresses. At the end of the distillation the distillate, containing the alcohol, boric acid, nitric acid, and lime, is transferred again to the crucible, evaporated to dryness at a very gentle heat, and ignited to a constant weight over the blast lamp. The greatest care is to be taken to secure similarity of conditions under which the crucible and lime are weighed before and after ignition, and definite periods of cooling before weighing—ten minutes is a suitable time—should be allowed to pass, in order to eliminate, as far as possible, the effect of atmospheric condensation upon the large surface of platinum.

The tendency of the process is to yield figures slightly larger than the truth, as was shown in the test analyses which are given in the full description of the process, to which reference has been made. In distilling 0.2 g. of boric anhydride and fixing it in 1.5

g. of calcium oxide, the mean error was shown to be about 0.0010+g. In the work upon the waters parallel determinations rarely differed by more than that amount.

Chlorine (with bromine and iodine.).—The precipitation of chlorine, together with bromine and iodine, was usually effected without difficulty by treating the weighed portion of water—from 100 g. to 500 g., according to the circumstances—with nitric acid to a slightly acid reaction and silver nitrate in excess, directly and in the usual manner, excepting that the operation was effected in the cold and that the whole was allowed to stand quietly in the dark for several hours to secure perfect subsidence of the precipitate. In only one or two cases did the silica, which the waters carry abundantly, give rise to trouble in the precipitation. In such cases comparative tests were made as just described upon portions treated previously with an excess of pure sodium hydrate, it having been noticed that certain very siliceous waters, which of themselves very quickly filled the pores of the filter with an invisible coating of silica, could be made to filter with rapidity by first treating them with sodium hydrate in excess, and then with nitric acid to slight acidity. Treatment with acid alone did not bring about the desired condition of the silica. In the case of the water of the Coral Spring the proportion of silica is so great and its character such that to secure satisfactory results we were obliged to convert the silica to hydrofluosilicic acid, by the addition of hydrofluoric acid and nitric acid, before precipitating the silver salt. The silver chloride (including the bromide and iodide) was collected upon asbestos in a perforated crucible, dried directly over the free flame of a Bunsen burner turned very low and at a temperature below its melting point, and weighed. In the case of waters containing hydrogen sulphide in quantity precipitable by silver nitrate the portion for analysis was rendered ammoniacal before the addition of the silver salt, and the precipitated silver sulphide was filtered off. The silver salts of the halogens were then precipitated from the filtrate by nitric acid, and the process was completed as described.

Silica, iron and aluminum, calcium, and magnesium.—A single weighed portion of water served for the determination of the silica, iron and aluminum, calcium and magnesium. The water

was acidified with hydrochloric acid, evaporated to dryness in platinum over the water-bath, again acidified and again evaporated. The residue was digested with hot water containing a little hydrochloric acid, and the solution was filtered from the residue of silica, which was ignited in platinum and tested as to its purity in the usual manner with hydrofluoric acid.

In the filtrate ammonia, in the slightest excess, precipitated ferric and aluminic hydrates from the boiling solution, and these were collected, ignited, and weighed as oxides in the usual manner. The lime was thrown out of the hot ammoniacal filtrate by ammonium oxalate, and the calcium oxalate filtered off after standing twelve hours in the cold. When the amount precipitated exceeded a few milligrams it was dissolved in hydrochloric acid, reprecipitated by ammonia and ammonium oxalate and collected upon a filter, the filtrate from the second precipitation being added to that from the first. The calcium oxalate was dried, ignited, and weighed as calcium oxide.

The filtrate (or the united filtrates) from the calcium oxalate was evaporated in platinum, and the residue was gently ignited to destroy the ammonium oxalate—which tends to delay the precipitation of the magnesia if permitted to remain in the solution—and dissolved in hydrochloric acid. From this solution the magnesia was precipitated in the usual manner by microcosmic salt and ammonia, and after standing twelve hours or more, the phosphate was collected upon asbestos in a perforated crucible, washed with the mixture of one part of ammonia to three of water, commonly used in the process, moistened at the end of the washing with a strong solution of ammonium nitrate in ammonia, ignited for a moment to redness, and weighed as the pyrophosphate.

Sodium, potassium, and lithium.—In some of the earlier work only sodium and potassium, of the alkalies, were determined in the natural water, and for the determination of the lithium portions of the concentrated water were treated by the method which will be referred to later. In the greater number of cases the same portion of the natural water served for the determination of lithium as well as sodium and potassium. The portion for analysis was acidified in platinum with hydrochloric acid and evaporated to

dryness. The residue was extracted with hot water slightly acidulated with hydrochloric acid, the solution was separated from the precipitated silica by filtration, and to the filtrate was added, with care to secure a sufficiency and yet to avoid a great excess, barium hydrate made from the nitrate, itself purified by precipitation by nitric acid. Filtration separated the precipitated hydrates of aluminum, iron and magnesium, with barium sulphate; and from the filtrate ammonium carbonate and ammonia precipitated, upon boiling, nearly the whole of the barium and calcium as carbonates. The filtrate from the precipitated carbonates was evaporated to dryness, and the residue was gently ignited to remove ammonium chloride, treated with hydrochloric acid, freed from the excess of acid by evaporation on the water-bath to dryness, dissolved in a very small amount of water, and treated again with a few drops of the solution of ammonium carbonate and ammonia. This treatment usually results in the precipitation of traces of barium carbonate which have previously escaped removal by the similar treatment of the larger volume of liquid. The filtrate from this last slight precipitate was again evaporated and treated as before; and, in case any further precipitation took place, the process was repeated until the purification was complete. Usually, however, the first precipitation in the larger volume of liquid and the second treatment in the solution reduced to small bulk are together sufficient to remove the barium carbonate completely. The filtrate from the last precipitate was evaporated in a weighed platinum dish, and the residue, consisting of the chlorides of sodium, potassium, aud lithium, was dried, gently ignited, and weighed.

From the mixed chlorides the lithium salt was separated by a process which, devised by one of us during the progress of the work and described elsewhere in full,[1] needs but brief outlining in this place. To the concentrated solution of chlorides amyl alcohol is added and heat is applied, gently at first to avoid danger of bumping, until, the water disappearing from solution and the point of ebullition rising and becoming constant for some minutes at a temperature approximately that at which the

[1] Bulletin of the U. S. Geological Survey, No. 42, p. 73; Am. Chem. Jour., vol. 9, p. 33. This Journal, 1, 185.

alcohol boils by itself, the chlorides of sodium and potassium are deposited, and the lithium chloride is dehydrated and taken into solution. At this stage of the proceeding the liquid is cooled, a drop or two of strong hydrochloric acid being added to reconvert traces of lithium hydrate possibly in the deposit, and the boiling is continued until the alcohol is again free from water. If the amount of lithium chloride present in the mixture of salts is small it is now found in solution; the chlorides of sodium and potassium, excepting the traces for which correction will be made subsequently, are in the residue; and the separation may be effected by filtration and the washing of the residue with amyl alcohol previously dehydrated by boiling. If, on the other hand, the weight of lithium chloride present exceeds ten or twenty milligrams it is advisible, though not absolutely essential to the attainment of fairly correct results, to decant the liquid from the deposit, wash the latter with a little anhydrous amyl alcohol, dissolve in a few drops of water, and repeat the separation by boiling in amyl alcohol.

The filtrates, first measured apart, and united with the washings and evaporated to dryness, and the residue treated with sulphuric acid in excess, ignited gently at first and finally to the fusing point, is weighed as the neutral lithium sulphate containing traces of the neutral sulphates of sodium and potassium. From the weight thus found the subtraction of [illegible] g for every 10 cm^3 of filtrate exclusive of washings makes the proper correction for the amounts of the sodium and potassium salts present, and the residual weight represents the pure lithium sulphate. In cases where the precipitate consists solely of sodium chloride, the correction upon the weight of the sulphate is [illegible] g; when potassium chloride constitutes the residue the correction amounts to [illegible] g for every 10 cm^3 of filtrate. The alcohol used in washing has, within reasonable limits, no appreciable solvent effect upon the precipitate.

The precipitate, consisting entirely of sodium chloride and potassium chloride, is ready for the balance after drying a few minutes directly over a flame turned low, if the filtration is effected by means of asbestos in the perforated crucible. If paper is used as the filtering medium after the solution of the precipitate the

evaporation of the solution and the gentle ignition of the residue. The weight of the insoluble chlorides actually obtained is corrected for the solubility of the salts in amyl alcohol by adding 0.00092 g. for every 10 cm^3. of amyl alcohol in the filtrates, exclusive of washings; 0.00041 g. of this representing the sodium chloride dissolved, and 0.00051 g. the potassium chloride. With due attention to the reduction of volume of the residual alcohol before filtering the correction may be held easily within narrow limits.

In applying this method to the analysis of the waters of the Yellowstone Park the practice of reducing the volume of amyl alcohol to about 15 cm^3. before filtering was followed uniformly. The corrections, therefore, were always the same, and amounted to an addition of 0.0007 g. to the apparent weight of sodium chloride, 0.0008 g. to that of potassium chloride, and a subtraction of 0.0017 g. from the weight of lithium sulphate indicated by the balance. In many cases the lithium present in the waters, though capable of detection by the spectroscope, amounted to so little in the portions devoted to its determination that the application of the correction left nothing to be called lithium sulphate. In such cases the lithium is counted as a "trace."

From the mixture of sodium chloride and potassium chloride the potassium salt was separated as potassio-platinic chloride. The chlorides were dissolved in a small amount of water, platinic chloride was added in solution, in quantity considerably in excess of the amount necessary to effect the conversion of all the potassium and sodium present to the double platinum salts, and the mixture evaporated on the water bath to the point at which the residue becomes pasty if removed from the bath and allowed to cool. The crust thus formed was drenched with alcohol of 80 per cent., digested in the cold until thoroughly disintegrated, filtered off on an asbestos felt, washed with alcohol of 95 per cent., dried at a gentle heat, and weighed.

The lithium and potassium having been thus found directly, the sodium is given, in the form of the chloride, by the difference between the sum of the lithium and potassium chlorides and the total weight of the three chlorides; or, by the difference between the weight of potassium chloride found and the weight of the chlorides of potassium and sodium found (the proper correction

having been applied) after the separation of the lithium. It may be urged against the former plan that to bring the three chlorides to a definite condition for weighing is a difficult undertaking in view of the great tendency of lithium chloride to exchange chlorine for oxygen in the presence of water. Our experiments indicated, however, that under repeated treatments by moistening and ignition the mixed chlorides did not change in weight materially, and we are disposed to attribute the result in part to the protective influence of the sodium and potassium salts upon the relatively small amount of lithium chloride, and in part to the care taken to secure thorough drying at a low temperature previous to the gentle ignition. In our work upon the waters we weighed the three chlorides together and found the sodium chloride by deducting from this weight the weights of potassium chloride and lithium chloride deduced by analysis.

A weak point in the process which we have employed in the determination of the alkalies would seem to be found in the separation of the magnesia by means of barium hydrate. In connection with this subject it is of interest to note that in the greater number of waters magnesia was present only in inconsiderable quantity, and further, that in the case of those in which it did appear in appreciable amounts the examination of the solution of the lithium sulphate, after the separation by amyl alcohol, failed to indicate the presence of magnesia in amount sufficient to be precipitable by microcosmic salt in an ammoniacal solution and under the usual conditions of the test, though this would be the place in which magnesia should appear if it had escaped removal from the chlorides. We are constrained to believe, therefore, that the process of separation which we adopted was exact within reasonable limits.

Ammonia and albuminoid ammonia. The determination of ammonia was effected in the usual manner by distilling a measured portion of water with sodium hydrate and nesslerizing the distillate. A second portion of water was distilled with sodium hydrate and potassium permanganate and the distillate was nesslerized. The former process gives the combined ammonia present in the water, the latter the total ammonia, which includes, beside the ammonia existing as such in combination, that ammonia which is

developed from other nitrogenious compounds by the action of the permanganate. The difference between the figures of the two processes is the albuminoid ammonia, so called. The composition of these waters renders them peculiarly liable to boil explosively, and some difficulty was experienced at first in the distillation; but the apparatus which is described in the paragraph on the determination of the boric acid proved to be of service in this case also, and made the determination practicable. The Nessler solution was very delicately adjusted, and the tests were made in small tubes and carried to the limit of 0.000001 g.

TREATMENT OF CONCENTRATED WATERS.

In the examination of the concentrated waters the whole sample was evaporated to dryness, and the residue thus left was extracted with hot water. It is usual in treating concentrated waters to add sodium carbonate during evaporation, and to wash the residue until the washings cease to show an alkaline reaction. Four of the waters of which we had concentrated samples were, however, so alkaline of themselves that the addition of sodium carbonate was superfluous, and all were so siliceous that to wash the residue of evaporation until it was free from alkali was a matter of the greatest tediousness and difficulty. We found it best and sufficient to wash only to the vanishing point of the spectroscopic reaction for lithium in the washings. In this manner the separation of the greater part of the silica with the insoluble constitutents of the water was effected without an entirely unreasonable expenditure of time and effort. The filtrate and washings were thoroughly mixed and made up to a definite weight. The residue was kept by itself for separate treatment.

The filtrate should contain the bromine, iodine, boron, arsenic, antimony; the copper, lead and tin, which can be present in the waters only in traces; the lithium, cæsium, rubidium, thallium, and part of the silicon and aluminum. The precipitate should contain the titanium, phosphorus, fluorine, iron, mangenase, barium, strontium, calcium, magnesium, with the remainder of the aluminum and silicon.

STRENGTH OF THE CONCENTRATED WATERS.

The degree to which the concentration of the waters had been

carried in the field was fixed by the determination of the total amount of halogens in a weighed portion of the extraction of the residue of evaporation. This portion was diluted, acidified with nitric acid, treated with silver nitrate until the precipitate ceased to form, and the last was collected on asbestos in a perforated crucible, dried (over the free flame of a Bunsen burner turned low) at a temperature considerably below its melting point, and weighed. Determinations of this sort were made in duplicate. Knowing the weight of the silver salt thus found and the weight of the same precipitable from a known weight of the natural water, the exact value of the concentrated water, in terms of the natural water which it represented, became known.

TREATMENT OF THE RESIDUE.

Fluorine.—The residue, dried by gentle heating, with the ash of the filter employed in separating and washing it, was placed in a retort of the pattern described in the section treating of the determination of boric acid in the natural water, drenched with strong sulphuric acid and heated to 150° C., while a slow current of dry air was forced through the emulsion and thence directly into a U tube partly filled with dilute ammonia. The fluorine contained in the water should appear in the residue as calcium fluoride, should be volatilized as silicon fluoride by the action of the sulphuric acid, and, if present in more than very small amounts, should be indicated by the precipitation of silica in the U tube containing ammonia. Proceeding in this manner we found in the waters which we examined no fluorine; and on treating the ammoniacal liquid of the U tube according to the method of Berzelius, with zinc oxide dissolved in ammonia, evaporating until the solution ceased to evolve ammonia, filtering, adding to the filtrate calcium chloride followed by sodium carbonate, boiling, igniting the precipitate and extracting it with acetic acid, we were not able to detect a residue of calcium fluoride; nor in the zinc oxide precipitated by the expulsion of the ammonia dissolving it could we discover silica.

Barium and strontium.—The contents of the retort were transferred to platinum, treated with hydrofluoric acid (purified by redistillation) in sufficient amount to volatilize all silica, and evapo-

rated to dryness with a little sulphuric acid. To insure thoroughness of action this process of treatment was repeated. The residue was fused with sodium carbonate and the melt was extracted with hot water containing a few drops of alcohol. The precipitate was separated from the solution by filtration and digested with hot dilute acetic acid, which should dissolve the barium, strontium, calcium, and magnesium carbonates. The solution in acetic acid, separated by filtration from the portion unacted upon, was nearly neutralized with ammonia, and to it was added in solution an amount of ammonium sulphate equal to fifty times the weight of the alkaline earths present, the whole being permitted to stand quietly twelve hours. By this treatment barium and strontium should be precipitated as sulphates, while calcium and magnesium should remain dissolved. The sulphates of barium and strontium may be separated afterward by digestion during twelve hours (at the ordinary temperature and with frequent stirring) in ammonium carbonate, the decantation of the supernatant liquid, the repetition of the treatment with ammonium carbonate, and, finally, the action of hydrochloric acid upon the residue—the strontium carbonate dissolving and the barium sulphate remaining insoluble in this acid. This mode of separating the barium and strontium was not actually applied in our work, inasmuch as we obtained from the quantities of water with which we had to deal no precipitate by ammonium sulphate, and it is mentioned only for the purpose of completing the scheme of analysis.

The solution which had been treated with ammonium sulphate to precipitate barium and strontium was preserved for subsequent examination for manganese.

Phosphoric acid.—The extract of the fusion in sodium carbonate was acidified with nitric acid, evaporated to small bulk, and from it the phosphoric acid was precipitated in the usual manner as a phosphomolybdate of ammonia. The precipitate, after standing twelve hours, was separated by filtration through a felt of asbestos in a perforated crucible, washed with the precipitant, and dissolved in ammonia. From this solution the phosphoric acid was precipitated by the magnesium and ammonium chloride mixture, and the ammonia-magnesium phosphate was filtered off on asbestos, washed with dilute ammonia (one part of ammonia to three of

water), moistened with ammonium nitrate in ammonia, dried, ignited at a red heat, and weighed as magnesium pyrophosphate.

Manganese.—The residue left, after the extraction of the fusion in the sodium carbonate with water containing a little alcohol and the treatment with acetic acid, was fused again in a little sodium carbonate, the melt was dissolved in the crucible by heating with strong sulphuric acid, and this solution was poured into cold water. In this manner was obtained a solution containing the titanium and iron, and a part of the aluminum and manganese. From it all these elements, excepting the last, were precipitated by adding ammonia to an alkaline reaction followed by acetic acid to faint acidity, and boiling. The precipitate was kept for examination later. The solution was mixed with that which had been treated with ammonium sulphate in the test for barium and strontium, and to the mixture, heated on the water bath, were added ammonia in excess and bromine water. The precipitate thus obtained was ignited and weighed as Mn_3O_4, it being so trifling that solution and reprecipitation as the phosphate was unnecessary.

Iron.—The precipitate of ferric, titanic and aluminic hydrates was fused in a little sodium carbonate, the fusion was dissolved in strong sulphuric acid, this solution poured into cold water, and the dissolved iron, titanium, and aluminum were separated as follows, by a process devised by one of us and described fully elsewhere.[1] Tartaric acid, amounting to three times the weight of the oxides of iron, aluminum, and titanium present, was added to the solution, which was then made faintly ammoniacal and submitted to the action of a current of hydrogen sulphide. The precipitated ferrous sulphide was filtered off (care having been taken that the solution should still be alkaline before filtering), washed with hot water sufficiently but expeditiously, dissolved in hydrochloric acid, and the iron, first oxidized by bromine, was precipitated by ammonia and weighed as the oxide.

Titanic acid.—The filtrate from the ferrous sulphide was acidified strongly with sulphuric acid, heated to the boiling point, and treated with potassium permanganate until its characteristic color appeared. The final products of the action of the permanganate upon the tartaric acid are carbonic acid and water, but formic acid

[1] Bulletin of the U. S. Geol. Survey, No. 27, p. 16; Am. Chem. Jour., vol. 7, 1885–'86, p. 283.

appears as an intermediary step in the oxidation. During the first action of the permanganate its conversion to manganous sulphate is rapid, but with the development of formic acid there takes place by its action upon the permanganate freshly added a precipitation of a brown hydrate of manganese which, redissolving readily so long as tartaric acid remains, yields only slowly to the further action of the formic acid. When, therefore, manganic hydrate appears abundantly in the boiling solution and does not dissolve perceptibly one may be reasonably certain that the conversion of the tartaric acid to at least the intermediary stage has been accomplished; but for the sake of greater security, the addition of the permanganate may be continued until the appearance of its color. When the amount of tartaric acid in the solution is known, as should be the case always, a simple mode of proceeding is, perhaps, to employ for the oxidation the amount of permanganate theoretically required. A weight of potassium permanganate amounting to two and a half times the weight of the tartaric acid to be destroyed is sufficient to carry the oxidation to the ultimate limit, providing there be no deposition of a manganic hydrate.

The residual permanganate and the insoluble hydrate were reduced and dissolved by the addition of a little ammonium bisulphite to the acid solution, and, the excess of sulphurous acid having been boiled off, acetic acid was added to the clear solution in quantity sufficient to make the amount of the absolute acetic acid present from 7 to 11 per cent. by volume; enough sodium acetate was introduced to fix all the sulphuric acid as sodium sulphate, and the solution was boiled. If titanium is present it is precipitated under these conditions, while the aluminum remains dissolved. If the quantity of the precipitate thus obtained is considerable, it should be filtered from the liquid in which it floats and purified from the traces of manganese which it may carry, by fusion in a small quantity of sodium carbonate, the solution of the melt in sulphuric acid, and reprecipitation by ammonia.

In the waters which we examined we found no precipitate of titanic hydrate at the point in the process where it should appear were titanium present.

TREATMENT OF THE AQUEOUS SOLUTION.

The aqueous solution, its degree of concentration having been determined in the manner already described, was divided into five portions. Two of these were taken for the determination of bromine, iodine, lithium, cæsium, rubidium, and thallium, in duplicate; two for the determination in duplicate of arsenic, antimony, tin, copper, lead, and boron; and the remaining portion was reserved to be used in an emergency.

Iodine and bromine.—The portion for the determination of the elements of the former group was weighed and evaporated to a pasty condition. The residue was thoroughly extracted with strong alcohol, treated with water reduced again nearly to dryness, and again extracted with alcohol. The extract was evaporated after the addition of a drop or two of a strong solution of sodium hydrate, and this residue was extracted with alcohol like the former residue. The residues of the extractions with alcohol were kept for subsequent treatment. The alcoholic solution containing presumably all the iodine and bromine, with a small proportion of the chlorine originally in the water, was evaporated to dryness, and the solution of the residue in water was acidulated with sulphuric acid mixed with a solution of ferric sulphate introduced into a retort of the pattern previously described and figured in connection with the determination of boric acid in the natural waters, and distilled. The distillate was collected in a U tube attached to the condensing tube and sealed with carbon disulphide and water. If iodides are present in the retort to the minutest degree the iodine is set free as such, its characteristic color appears in the seal of carbon disulphide, and its amount may be determined by titration in the usual manner with sodium hyposulphite.

In the waters which we examined iodine was not present in sufficient quantity to appear in the test of the amounts at our disposal.

To the liquid remaining in the retort after the distillation had been prolonged sufficiently to insure the complete volatilization of the iodine a few crystals of potassium permanganate were added, and the distillation was continued. This distillate was collected as before in a U tube joined to the condenser, but the

seal was made with chloroform and water. At the beginning of the operation the seal of the U tube showed in most cases the characteristic color of bromine, but this was discharged before the end of the operation by the excess of chlorine and replaced by the color of the latter. The contents of the U tube were transferred to a beaker and treated with pure sodium hydrate and zinc, and from the solution of sodium bromide and chloride thus obtained the silver salts were precipitated by silver nitrate, in the usual manner, after acidifying with nitric acid. The silver bromide and chloride thus precipitated were collected together upon asbestos in a perforated crucible, dried, weighed, and dissolved in potassium cyanide. From the cyanide solution the silver was deposited by electrolysis, according to the process described elsewhere by one of us for the indirect estimation of bromine in the presence of chlorine.[1] In this manner we obtained the weight of the mixture of silver chloride and silver bromide, and the weight of the silver contained in the mixture. The amounts of bromine and chlorine in the mixed salts were determined from these data by the solution of a single algebraical equation.

Lithium.—The contents of the retort after the distillation of the bromine were united with the residues of the extractions by alcohol, the excess of sulphuric acid was volatilized, the residue dissolved in hydrochloric acid, the sulphates converted to chlorides by means of barium chloride, the baric sulphate thus formed separated by filtration, alumina, silica, and barium carbonate precipitated by ammonium carbonate and separated by filtration, and the solution evaporated to dryness. The residue was gently ignited to remove ammonium chloride, dissolved in water acidulated with hydrochloric acid, and this solution, concentrated until just ready to deposit, was drenched with strong alcohol. The sodium chloride precipitated was washed with alcohol until the spectroscope indicated entire freedom from lithium chloride. The alcoholic solution of lithium chloride was evaporated to dryness, and the residue was dissolved in a little water acidulated with hydrochloric acid. This solution was treated with a little pure barium hydrate (made from the nitrate precipitated from nitric acid) to remove traces of magnesia, and filtered, and from the

[1] Bulletin of the U. S. Geol. Survey, No. 42, p. 89; Am. Chem. Jour., vol. 8, p. 421.

filtrate the barium was removed by treatment with ammonium carbonate, repeated if necessary. From the solution of lithium chloride thus obtained the lithium was separated according to Mayer's method[1] by evaporation with hydro-disodic phosphate and sodium hydrate, the treatment of the residue with sufficient hot water to dissolve the salts, which should be soluble with the aid of gentle heat, the addition of an equal volume of ammonia, and digestion in the cold during twelve hours. The precipitate was filtered upon asbestos in a perforated crucible, washed with a mixture of equal parts of ammonia and water, ignited, and weighed. The filtrate from this precipitate was evaporated, and the residue of evaporation was again treated as before to recover traces of lithium; and a similar process of treatment was applied to the filtrate from the second precipitate. The three precipitates thus obtained contained presumably all the lithium as the tribasic phosphate.

This is the method of determining lithium which was employed previous to the development of the process which has been described in connection with the account of the determination of lithium in the natural waters (p. 16). Were the work to be repeated we should unquestionably employ the amyl alcohol process exclusively, although it may be said for the determinations by Mayer's method that their actual error was very much diminished by the use of comparatively larger portions of material for the analysis, and we are confident that the results are on that account fairly trustworthy.

Cæsium, rubidium, and thallium. The filtrate from lithium phosphate was freed from ammonia by boiling; the phosphoric acid was precipitated by ferric chloride and ammonia with the usual precautions to secure a suitable proportion of the iron salt and to keep the excess of ammonia at the lowest possible limit, and removed by filtration; the filtrate from ferric phosphate and ferric hydrate was evaporated to dryness and gently ignited to remove ammonium salts; the residue thus left was dissolved in water and treated with platinic chloride in excess of the amount necessary to convert all the alkaline chlorides present to platinum salts.

[1] [illegible]

The precipitate by platinic chloride was extracted by boiling repeatedly with small portions of water, and the residues of this treatment were tested from time to time spectroscopically. The characteristic lines of cæsium and rubidium appeared more distinctly toward the end of the process of extraction when they were visible at all. Thallium, which should appear, if present, in the intermediate extracts, failed to show itself in any of the waters.

Arsenic, antimony, tin, copper, and lead.—The weighed portion of the concentrated solution was acidified with hydrochloric acid, heated to 70° C., and submitted to the action of a current of hydrogen sulphide during several hours. The precipitated sulphide was filtered off upon anthracene in a perforated crucible, following the method described by one of us.[1] The crucible with the adherent filter of anthracene and the precipitate were placed in a small beaker, drenched with benzol, and gently warmed until the anthracene dissolved. Water containing sodium hydrate was added and, after sufficient digestion on the water-bath, the liquid and precipitate were transferred to a paper filter, previously moistened with water. In this manner the aqueous solution, carrying the arsenic, antimony, and tin, if present, was permitted to traverse the filter, while the sulphides of copper and lead, with possibly free sulphur, remained with the benzol upon the filter. After thorough washing with water the receptacle for the filtrate was changed, the filter was moistened with alcohol, and thus the benzol, with the anthrazene in solution, was induced to pass through the paper, which, with the residue upon it, was washed with benzol followed by alcohol and then by water. The filter was carefully incinerated, and the ash was digested with a few drops of nitric acid. The solution thus obtained was evaporated to dryness, the residue was moistened with hydrochloric acid, and, after the volatilization of the most of the acid, dissolved in hot water. From this solution hydrogen sulphide should precipitate copper and lead as sulphides. In the single case of that water which had been concentrated in porcelain no black sulphide was obtained at this point in the operation; in other cases, a trifling black precipitate fell, which subsequent examination

[1] Bulletin of the U. S. Geol. Survey, No. 42, p. 86; Am. Chem. Jour., vol. 7, 1885-'86, p. 87.

proved to be copper sulphide exclusively, doubtless originating in the concentrating vessel.

The second precipitation with hydrogen sulphide was introduced to test the presence of copper and lead, in the process just outlined, because the residue left upon the filter when the benzol is washed from it is apt to be dark in color, even in the absence of black sulphides.

The alkaline filtrate from the benzol and the insoluble residue were acidified with hydrochloric acid, after the addition of a drop or two of ammonium sulphide; the precipitate was filtered off on asbestos in a perforated crucible; the filter and precipitate were dried and treated in a small beaker with fuming nitric acid; the solution in nitric acid was evaporated to dryness, the residue was dissolved in hydrochloric acid, and from this solution, made strongly alkaline with ammonia containing some ammonium sulphide (the last to keep tin, if present, in solution) but kept small in volume, the arsenic was precipitated by the magnesium chloride mixture. The liquid was briskly stirred and then allowed to stand quietly for twelve hours at least. The precipitate was transferred by means of the mother liquor, without the use of other liquid, to a perforated crucible carrying an asbestos felt, washed with a mixture of one part of ammonia to three of water, dried carefully, ignited in an atmosphere of oxygen, and weighed as magnesium pyroarsenate.

The filtrate from the ammonia magnesium arsenate, which should contain the antimony and tin of the water, was evaporated to remove the great excess of the ammonia and acidified with hydrochloric acid. The sulphur thus precipitated should carry with it the antimony and tin as sulphides. In no case were we able to detect antimony in this precipitate. In one or two cases there were evidences of the presence of tin, but this doubtless came from the lining of the vessel in which concentration was effected; in the water which was concentrated in porcelain no tin was found.

Boric acid. The filtrate from the original precipitate by hydrogen sulphide in the acid solution was warmed gently, submitted to the action of a current of air to blow out the excess of hydrogen sulphide, brought nearly to neutrality by the addition

of sodium carbonate, made faintly alkaline with ammonia, filtered from the precipitate thus thrown down, and evaporated nearly to dryness after the addition of enough sodium hydrate to expel the ammonia and yet preserve strong alkalinity. The residue was acidified with hydrochloric acid and extracted thoroughly with alcohol, and the alcoholic solution was made alkaline with sodium hydrate and evaporated to dryness. This residue was dissolved in water, just acidified with hydrochloric acid, and from the solution the last traces of silica, alumina, lime and phosphoric acid were removed by the action of ammonia and ammonium carbonate, and filtration. In the filtrate the boric acid was determined according to Marignac's process, by treatment with magnesium chloride, ammonium chloride, and ammonia, evaporation to dryness, ignition at a red heat, extraction with boiling water, and the repetition upon the extract of the process of evaporation, ignition, and extraction. The residues left after extraction were together ignited and weighed, and the magnesia in the weighed material was determined by precipitation as the ammonio-magnesium phosphate and weighing as the pyrophosphate. The difference between the weight of the residue and its contents in magnesia is boric acid.

This was the process which we employed in the earlier determinations. The process which was developed later, and applied to the determination of boric acid in the natural waters, is preferable.

STATEMENT OF THE RESULTS OF ANALYSIS.

In stating the facts deduced from the analysis of the waters, it has seemed best to adopt the plan of recording the oxygen acids as anhydrides, the basic elements as such, and the oxygen which must exist in combination with the bases by itself; thus avoiding an arbitrary division of the basic elements among the oxygen acids and halogens. The basic oxygen, so called, of the alkaline waters was found by computing the amount of oxygen which would exist in the oxides of the basic elements and deducting from this amount the weight of oxygen equivalent to the halogen discovered by analysis. In the case of waters which carried such an excess of acid that the halogens may be supposed to be uncombined with bases, the basic oxygen is the amount of oxygen which

would exist in the oxides of the basic elements. In waters of such acidity that a portion only of the halogen may be supposed to be in combination with basic elements the equivalent in oxygen of the amount of halogen which is regarded as in combination was deducted from the total amount of oxygen of the oxides in computing the basic oxygen. The halogen which remains uncombined with the basic elements was, as a matter of course, assumed to be in association with hydrogen.

The hypothetical combination of the elements found in the waters must necessarily be largely imaginary, for nothing is more plainly evident than the impossibility of stating authoritatively, in the present condition of knowledge, the actual division of a number of acids among a number of bases in solution. This we do know, that the combination is determined not only by the relative proportions of all the constituents, but by the absolute quantities of all associated in a definite amount of liquid. It is with no purpose, therefore, of attempting to represent actual conditions of combination, but only for the sake of securing a common ground of comparison among themselves and with other waters that we have assumed arbitrarily the existence in these waters of the definite and simple combinations of the following scheme:

In alkaline waters, bromine has been combined as KBr; B_2O_3 as $Na_2B_4O_7$; As_2O_3 as $NaAsO_2$; P_2O_5 as Na_2HPO_4; chlorine has been assigned to the bases in the order NH_4, Li, K, Na; SO_4 has been linked with such bases as remain in the order NH_4, Li, K, Na, Mg, Ca; residual bases have been classed as neutral carbonates in the order Mg, Ca, Na; residual sodium has been represented as Na_2SiO_3; aluminum and iron have been put down as Al_2O_3 and Fe_2O_3; and residual CO_2 and SiO_2 have been shown as such.

In making up the hypothetical composition of acid waters the same general arrangement has been kept as far as possible in order to secure ready comparison with the alkaline waters. Though the relative avidity of hydrochloric acid is greater at ordinary temperatures than that of sulphuric acid, we have nevertheless represented hydrochloric acid as the free acid whenever it became necessary to represent one of them as free.

The sulphuric acid indicated by the analysis has been com-

bined with the bases in the order Ca, Mg, Al_2, Fe_2, Na, K, Li, NH_4; the residual bases have been combined with chlorine in the same order; the anhydrides B_2O_3, As_2O_3, P_2O_5, SiO_2, CO_2, and residual SO_3, have been stated as such; residual chlorine has been represented as HCl, and H_2S as such.

It is apparent that in some instances combinations have been imagined which might exist under some of the circumstances encountered, but which are improbable under other conditions met with in waters of a generally similar character; thus, for the sake of uniformity, we have represented the boric acid of the alkaline waters invariably as $Na_2B_4O_7$, and the phosphoric acid as Na_2HPO_4, regardless of the variable action of an excess of alkaline carbonate upon these salts in hot solutions and under prolonged digestion. So, also SiO_2 has been stated as free, when, if so, it must have been in solution in the presence of the carbonates of sodium, magnesium, and calcium; the neutral carbonates have been represented as such in the presence of free carbonic acid, both in hot and in cold waters; and potassium has been arbitrarily associated with bromine; and ammonium, lithium, and potassium have been preferred to other bases in combining the chlorine.

HAMPE'S METHOD OF DETERMINING Cu_2O IN METALLIC COPPER.*

BY FRED. P. DEWEY.

Having occasion to make a series of determinations of Cu_2O in copper in some suites of specimens in the National Museum, representing the process of refining pig copper to tough pitch metal, the Hampe† method was selected as being the best so far proposed.

The method is based upon the reaction between Cu_2O and $AgNO_3$, discovered by H. Rose,‡ and consists in treating the material to be analyzed with neutral solution of $AgNO_3$ in the cold for several days, when, according to Hampe, the following reaction takes place between Cu_2O and $AgNO_3$: $3Cu_2O+6AgNO_3$

*From Proceedings U. S. National Museum, 1888.

†Zeit. f. Berg-, Hütten- u. Salinen-Wesen, 1873, Vol. 21, p. 218.

‡Pogg. Ann., Vol. 101, p. 513.

$+3H_2O=4CuO, N_2O_5 3H_2O+2CuN_2O_6+6Ag$, whereby two-thirds of the copper is converted into an insoluble basic nitrate, and remains with the silver precipitated, while one-third goes into solution as normal nitrate. On filtering off the residue and determining the amount of copper contained in it, we can easily calculate the percentage of Cu_2O in the metal. The following series of results, calculated on Hampe's formula:

Cu_2O in Cu.

No. 1.	0.136	0.140	2.542	3.862	8.209
No. 2.	0.222	0.128	2.537	3.897	7.897

showed the method to be quite satisfactory as far as the agreement of duplicates goes.

Rammelsberg* investigated the matter slightly and found that only 28.80 per cent. of the copper went into solution. He assigned the following somewhat unusual formula to the insoluble nitrate: $Cu_{10}N_6O_{35}$. Hampe† has replied that Rammelsberg's investigation was too limited, being confined to a single determination, and proper precautions were not taken, while his own work was very elaborate and extended, and his deductions based upon many determinations. Rammelsberg used pure Cu_2O; Hampe used both Cu_2O‡ and metallic copper containing a known amount of Cu_2O, but with the latter material the amount of Cu_2O present was so small that the unavoidable errors of determination would completely mask the difference between 28.8 per cent. and 33.33 per cent.

Since, however, in the actual use of the method for determining Cu_2O in copper there is always a large excess of uncombined copper present which might possibly affect the reaction, it seemed desirable to take up the subject for further investigation.

That the free copper influences the reaction is readily shown by the fact that the filtrate from the residue of Ag and $H_6Cu_4N_2O_{12}$ contains much nitrite of copper.

More recently Wells|| in a different connection has fully estab-

*Ber., 1877, p. 1780.

†Ztschr. J. anal. Chem., Vol. 17, p. 127.

‡Hampe's Cu_2O always contained CuO.

||H. L. Wells and S. L. Penfield. Gerhardite and Basic Cupric Nitrates, the chemical work by the former. Am. J. Sci., Vol. 30, p. 56.

lished the Hampe formula as representing the reaction between pure Cu_2O and $AgNO_3$.

In all ordinary cases, such as the determination of Cu_2O in tough pitch metal, the determination of Cu_2O in the native copper of Lake Superior by Monroe,* and the test cases, using Cu containing Cu_2O, of Hampe, mentioned above, the unavoidable errors of the work are so great that it is immaterial whether the constant of solution is 28.8 or 33.33, but in the case of the series I had under investigation, where in one sample 8 per cent. of Cu_2O was found by the Hampe constant, such a difference could not be tolerated.

For the purpose of this investigation, therefore, a series of samples was prepared corresponding exactly with the material to which the method would be applied; that is, metallic copper containing Cu_2O, but containing a large amount of Cu_2O. It was necessary that the samples should contain nothing else.

A tolerably pure $CuSO_4$ was dissolved in hot water till a strong solution was obtained; it was, however, neither boiling nor saturated. This solution was cooled with agitation, and a crop of small crystals obtained. These were separated from the mother liquor, redissolved, and the operation repeated twice. The final crop of crystals gave an absolutely pure $CuSO_4$.

A solution was made of the pure crystals, and the copper precipitated by the electric current. In order to obtain the copper in suitable form, the plan used in refining copper commercially by the current was adopted. A strip of ordinary sheet copper was coated with paraffiine wax, and then graphite was sprinkled on it; this prepared strip was used as the first cathode. When a thin film of copper had deposited, the strip was taken from the bath, and the film of pure copper removed, the coating of wax making this quite easy to do. The thin sheet of pure copper was then used as the cathode, and the electrolysis of the solution continued, until a suitable amount of copper had been precipitated.

In this way some 200 grams of absolutely pure copper were prepared.

This pure copper was next melted in a scorifier, in a gas muffle, and allowed to absorb O from the air. In this operation the

*Trans. A. I. M. E., Vol. 8, p. 414.

greatest care had to be exercised to avoid contamination of the metal, especially by Fe. I am greatly indebted to Dr. H. G. Torrey, assayer of the United States Mint at New York, for making these fusions for me with such great care that only small amounts of Fe could be found in the metal after fusion.

55.853 grams of the pure metal were melted in a scorifier and allowed to absorb O until it had covered itself with the melted oxide; the resulting button, freed as much as possible from the melted oxide, weighed 45 grams.

Drillings from this button were crushed to an approximately uniform size, thoroughly mixed, marked off into squares, and samples for analysis made up by taking some from each square.

0.8318 gram were dissolved in HNO_3, converted into sulphate, and the copper precipitated by two Bunsen cells, yielding 0.8162 gram copper or 98.12 per cent.

2.0795 grams treated in this way yielded 2.0400 grams copper or 98.10 per cent. In the solution after the precipitation of the copper from the latter were found 0.00028 Fe=0.013 per cent.

2.7428 grams, determining the amount of copper going into solution, as well as that made insoluble by treatment with $AgNO_3$, yielded a total of 2.6918 grams copper=98.14 per cent.

The average of these three copper determinations is 98.12 per cent., adding to this the Fe, we have 98.135 per cent.; subtracting this from 100, we have 1.867 as the percentage of O present. 1.867 per cent. O=16.67 per cent. Cu_2O. We have therefore the composition of this material:

	Per cent.
Cu	83.317
Cu_2O	16.670
Fe	0.013
	100.000

For the Cu_2O determination absolutely pure $AgNO_3$ was dissolved in H_2O using 100 cc. H_2O to 3 grams $AgNO_3$. The Cu containing Cu_2O was weighed out, and the cold solution added, using 200 cc. or 6 grams $AgNO_3$ for each gram of the material. For the first few hours the mixture was repeatedly stirred, after which it was allowed to stand, with occasional stirring, for three to four days, being kept in a cool place all the time.

The residue, consisting of basic nitrate of copper and metallic silver, was filtered and thoroughly washed. It was then treated with quite dilute H_2SO_4, avoiding an excess. This dissolved the copper and, owing to its fine state of subdivision, a small portion of the silver also. The solution was filtered, evaporated, the silver precipitated by HCl, filtered again, and evaporated till fumes of H_2SO_4 were given off. The residue was taken up by H_2O, the solution filtered, and the copper precipitated by the battery, using two Bunsen cells, coupled zinc to zinc, and carbon to carbon. The following results were obtained :

	Per cent.
2.7081 grams gave 0.3507 Cu	=12.95
2.7428 grams gave 0.4355 Cu	=15.88
2.1279 grams gave 0.3106 Cu	=14.59
Average	14.47

Multiplying this by the Hampe constant, we have 24.44 as the per cent. of Cu_2O that should be present according to his formula, but the direct oxygen determination shows only 16.67 per cent. to be present.

If we assume that all the copper present as Cu_2O in the metal was transformed into the insoluble nitrate by the action of the $AgNO_3$ we have 14.47 per cent. Cu=16.29 per cent. Cu_2O, which is sufficiently close to 16.67 per cent., as found by the O. determination, to warrant the assumption as being true.

Thirty grams of the above material were mixed with an equal weight of the pure copper, and the mixture thoroughly melted, when it was removed from the muffle.

The method of examination was enlarged and slightly modified in certain points suggested by the work upon the first sample, with the hope of obtaining more closely concordant results.

The drillings were crushed fine and thoroughly mixed, then the whole was spread out and marked off into twelve squares, and four of these squares were taken for each analysis. Especial attention was given to securing uniformity in the samples weighed out, since some of the differences in the first set of results are due to differences in the samples taken.

The silver separated by the reaction was determined, and the iron remaining in the solution after the separation of the copper was also determined. The following results were obtained:

Sub.taken	Insol Cu.	Sol. Cu.	Fe.	Ag ppt.	Total Cu. Per ct.	O. Per ct.	Fe. Per ct.	Insol.Cu. Per ct.
3.0846	0.3406	2.6976	trace.	9.4538	98.496	1.504	trace	11.042
3.3337	0.3768	2.9062	. .	10.1969	98.509	1.491	. .	11.300
3.2479	9.3521	3.8479	0.00007	9.9700	98.525	1.475	0.002	10.840
				Average,	98.507	1.493	. .	11.06

Per cent. Per cent.
1.493 O = 13.33 Cu_2O
11.06 Cu = 12.455 Cu_2O

Dividing the total amount of silver precipitated, 29.6207 grams, by the total amount of material taken, 9.6662 grams, we have 3.0643 grams as the amount of silver precipitated by 1 gram of the material. Theoretically, 1 gram of a mixture containing 86.67 per cent. copper and 13.33 per cent. Cu_2O should precipitate 3.1544 grams silver, thus showing a deficiency of 0.0901 gram in the amount of silver precipitated, which indicates that 2.645 per cent. of the copper entered into the reaction without precipitating a corresponding amount of silver. Expressed atomically, we have:

Cu present as Cu_2O . . .	$11.837 \div 63.4 = 0.1867$	4.477
Cu not precipitating silver,	$2.645 \div 63.4 = 0.0417$	1

While these results are not as close as could be desired, yet considering the difficulties in the case arising out of the fact that all the experimental errors are thrown upon the 1.493 per cent. of O, they show that for every 4Cu in the Cu_2O entering into the reaction, one Cu did not reduce its corresponding amount of Ag. Taken in connection with the discovery of nitrate of copper in the filtrate from the silver and basic nitrate, these figures clearly establish the reaction as follows:

Reaction on treating copper containing Cu_2O with $AgNO_3$ in neutral solution.

$$4Cu_2O + Cu + 6AgNO_3 + 6H_2O = 2H_6Cu_4N_2O_{12} + CuN_2O_4 + 6Ag$$

Five grams of the first fusion were mixed with 55 grams of the pure copper and thoroughly fused. Treated in the same way as the second sample, this material yielded the following results:

Sub.taken.	Insol Cu.	Sol. Cu.	Fe.	Ag ppt.	Total Cu.	O.	Fe.	Insol.Cu.
					Per ct.	*Per ct.*	*Per ct.*	*Per ct.*
3.2626	0.2471	2.9801	0.000490	10.3304	98.915	1.070	0.015	7.58
3.5278	0.2781	3.2092	0.000455	11.1430	98.890	1.097	0.013	7.88
3.3826	0.2966	2.0479	0.000560	10.6036	98.873	1.110	0.017	8.77
				Average,	98.893	1.092	0.015	8.08

Per cent. *Per cent*

1.092 O=9.75 Cu_2O

8.08 Cu=9.10 Cu_2O

The composition of the material is therefore:

Per cent.

90.235 Cu

9.750 Cu_2O

0.015 Fe

100.000

Performing the same calculations upon these figures as in the preceding case we have:

Silver precipitated by 1 gram of material.

	grams.
Calculated	3.2217
Found	3.1531
Deficiency	0.0686

0.0686Ag=2.01 per cent. copper not precipitating Ag.

Cu present as Cu_2O	8.658÷63.4=0.1366	4.309
Cu not precipitating silver,	2.01 ÷63.4=0.0317	1

The conclusion arrived at from these figures is that, when copper containing Cu_2O is treated with a neutral solution of $AgNO_3$ in excess in the cold, all the Cu_2O is converted into the insoluble basic nitrate $H_6Cu_4N_2O_{12}$.

A NEW SEPARATING TUBE FOR FRACTIONAL DISTILLATIONS.

BY EDWARD HART.

I have tried a great many experiments within the two or three years just past, having in view the improvement of the apparatus for fractional distillation. The Le Bel-Henniger tube, while it is a very efficient apparatus, has two serious disadvantages; these

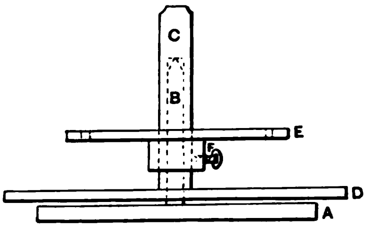
are, its costliness and fragility. Besides this, it cannot well be used for the separation of substances of boiling point over 150°, as the condensation becomes too great for the reflux tubes, and it is almost impossible, owing to its complicated form, to protect it by asbestos or other non-conducting material. The straight tube is not open to these objections, but the difficulty here is to get a proper return current of liquid flowing back into the flask, unless

the tube be left entirely open, when the separation is very incomplete. Filling the tube with round or irregular bodies does not answer the purpose unless we stop the distillation from time to time long enough to allow the liquid to run back. I have tried glass tubes, endwise and sidewise, wire gauze, and various other arrangements, but without marked success, until it occurred to me: why not have the return tubes inside the separating tube instead of outside as in the Le Bel-Henniger apparatus? After trying several ways of effecting this I have come to the conclusion that the arrangement shown in the figure is about the best. The large tube may be of any convenient length, and is not limited to any particular diameter. The one I have in use is about 19 inches long, nearly an inch in outside diameter, and the smaller tube below has a diameter about half as great. The central funnels catch any condensed liquid and return it through the central tubes, dripping from one funnel to another. The funnels should slip into the large tube easily without having too much play. The arrows show the course of the vapor. For high boiling liquids the tube is protected by a sheet of asbestos paper wired around it. The apparatus is easily made, can be readily cleaned, and is not fragile. I have used 90 per cent. commercial benzene (C_6H_6) for comparison, and find that my tube gives 1030 gms. out of 2560 gms. below 85°, while a Le Bel-Henniger tube of 4 bulbs gives 908 gms. below 85° of the same mixture.

A ROTARY FILTER STAND.

BY FRANK JULIAN.

The straight rack generally used for filtration without pressure has the disadvantage of occupying a large space on the work-table, and of not covering the beakers, etc., sufficiently to keep out dust. These are avoided in the circular stand here described, which I have found very convenient and effective. The dimensions given are for eight funnels, but the size may be increased with advantage. It should be made of well seasoned wood, and oiled or varnished.



The base A, 18 in. diameter by 1 in. thick, carries the post B, pointed at the top, 1 in. diameter by 9½ in. high from A. Over B fits the cap C, 12 in. by 2 in., to the bottom of which is fastened the plate D, 22 in. by ¾ in. The funnel-rack E, 14 in. by ½ in., having eight holes each ½ in. diameter and 5 in. from C, is fixed to the ring F, 3½ in. by 2 in., fitted with the binding-screw G. The cap C should revolve freely on B, which is kept coated with tallow. Stands of the above size are manufactured by E. H. Sargent & Co., Chicago.

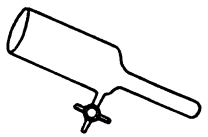
A NEW FORM OF ADAPTER.

BY CHARLES W. MARSH, PH. D.

In the distillation of liquids which are liable to bump up or to boil over into the tube in connection with the condenser or into the neck of the retort, much trouble is experienced in cleaning the connections and the condenser. The liquid is distilled until the distillate washes the condenser free from the impurities carried over, and the distillate which is thus contaminated by some of the contents of the retort jumping over and not distilling over, is put back into the retort to be redistilled. The distillate has merely washed the interior of the condenser and connections with the neck of the retort. This operation often takes much time, as is well observed when the liquid is colored and tolerably thick.

To remove the possibility of the condensing tube becoming soiled from contact with any substance that may spatter over, I

have introduced a glass stop cock on one side of the common adapter and at the angle where the large and small ends meet. By this means any impurities may be drawn off in the adapter and not traverse the entire length of the condenser.



Dimensions: Entire length, 23 cm.; length of large end, 11 cm.; length of small end, 11 cm.; internal diameter of large end. 40 mm.; internal diameter of small end, 13 mm.

The adapter is made of glass a trifle over 1 mm. thick. The diameter of the large end is made quite large to admit of general use.

I have tried the efficiency of the adapter by distilling water and alkaline potassium permanganate used in water analysis, and although some colored liquid would be caught in the angle of the adapter, still after drawing this off and washing it out and again drawing off, a distillate was obtained at the lower end of the condenser which was absolutely colorless. This form of apparatus may be found of value in the distillation of albuminoid ammonia according to Wanklyn. A second solution was boiled which consisted of an alkaline liquid with a copious precipitate. This was colored by means of fluorescein to better test the working in the distillation. By similar treatment a distillate free from color was obtained, although some of the liquid had been carried over into the adapter.

In certain lines of work it might be advisable to have a slight depression formed on the lower side of the neck of a retort, with a stop cock in the centre of this depression, the neck of the retort playing the part of adapter, as in the preceding cases

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[CONTRIBUTIONS FROM THE LABORATORY OF THE U. S. DEPARTMENT OF AGRICULTURE.]

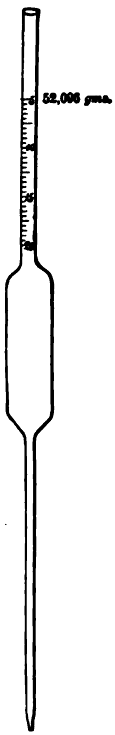
A CONVENIENT FORM OF PIPETTE FOR MEASURING OUT BEET, CORN AND SORGHUM JUICES FOR POLARIZATION.

BY C. A. CRAMPTON.

In Bull. No. 15, Chem. Div. U. S. Dept. Agr.,* I have described the method of procedure followed by the analysts at the

*Also in Chem. News, **55**, 207, this Journal, **1**, 339.

stations of this Department in the analysis of juices. For polarization it has been the custom to measure out a sample of juice, as it would require too much time to weigh, and for this purpose it has been the custom to measure out 50 cc. with a pipette, making subsequent correction of the polarization for the specific



gravity of the juice. When the polariscope used is one that has the factor 26.048, that is requires 26.048 gms. of pure sugar, dissolved and made up to 100 cc. of water to read 100 on the scale, it will be seen that 50 cc. of juice gives a weight in grams very nearly twice the normal amount to be taken for the instrument, or 52.096. Last year I had some pipettes made, which were graduated in the stem from 48 to 54 cc., with subdivisions of .5 cc., to be used in measuring out directly the correct amount of juices of different densities, to give double their polarization; the number of cc. to be used for each density to be ascertained from a table. These pipettes were used during the present sugar campaign in Louisiana by Mr. G. L. Spencer, at Magnolia Station, who found them very convenient. He has calculated a table to be used in connection with them.

I have had made recently a modification of this pipette, which does away with the necessity of a table, and which is illustrated in the accompanying cut. It is graduated from 48.1 to 51.1 cc., the subdivisions, being in tenths of a cubic centimeter, but the divisions are marked instead with degrees Brix, having a range from 5° to 20° each, .1 cc. division corresponding to .5° Brix. That is, after the degree Brix of a juice has been ascertained, the pipette, when filled up to the corresponding mark on the stem, will deliver 52.096 grams of the juice, (within a few milligrams, which is as close as it is possible, to weigh it out.) So it is necessary to divide the polarization by two, or to read the solution in a 400 mm. tube, to give directly the per cent of sucrose in the juice.

The calculation for the graduations is taken from Stammer's table for specific gravities and corresponding degrees Brix. For

commercial work these pipettes will weigh out the juice with sufficient accuracy, and I think they will prove convenient for rapid work, and save considerable time in the calculation of results.

QUANTITATIVE ESTIMATION OF ADULTERANTS IN LARD.

BY H. W. WILEY.

The progress of qualitative analysis has rendered it possible for the skilled analyst at the present time to detect with certainty every commercial adulteration of lard. It is probably true that laboratory adulterations amounting to only 2 or 3 per cent. may escape the quest of the skilled chemist ; but when adulterations are made for commercial purposes the amount of adulterant added is always in sufficient quantities to render its qualitative detection easy. For quantitive purposes, however, the matter is not so readily determined. For practical purposes the two adulterants which are used in making compound lards are cotton oil and the stearines derived by pressing partially crystallized lard or tallow. The first of these stearines is usually called prime lard stearine and the second oleostearine. The following methods have been proposed for the quantitative detection of these adulterants:

1. The amount of insoluble residue obtained upon treating the samples with a mixture of ether and alcohol. This method, as was shown in the celebrated trial of McGeoch, Everingham & Co. against Fowler Bros. in Chicago, was wholly unreliable, and it may be dismissed from the category of useful methods.

2. *The specific gravity.*—This method has great value and may be relied upon to give approximate results.

3. *Absorption of iodine.*—This method would be an excellent one for determining the amount of cotton oil added to a lard provided that no stearine was present ; but the careful addition of cotton oil and stearine will enable the mixer to make a lard whose iodine number is almost identical with that of the pure article.

4. *The rise of temperature which samples undergo when mixed with sulphuric acid.*—This process divised by Maumene may give valuable information in regard to the quantity of adulterants

added. Unfortunately, however, it is open to the same objection as lies against the iodine method, viz: the rise of temperature with the stearine is much less than that with lard so that by the addition of stearine and cotton oil a mixture can be made in which the rise of temperature is not very much greater than that produced by lard itself.

5. *Melting point of the samples.*—This method is of but little practical benefit when taken alone. Mixtures of lard and cotton oil do not show a mean melting point as indicated by theory. Cotton oil melts one or two degrees below zero while pure lard melts at about 40°. A mixture of equal quantities of these two substances shows a theoretical melting point of 20°. Such a mixture, however, will melt only a few degrees below the fusing point of pure lard.

6. *Refractive index.*—I am not aware that the refractive index has been used as a method of quantitatively approximating the amounts of cotton oil and stearine added and I propose to say a few words in regard to its value in this respect. Fourteen samples of lard known to be pure hog grease examined in my laboratory gave the following mean data:

Refractive index	1.4620
Percentage of iodine absorbed	62.48
Specific gravity at 35°	.9053
Rise of temperature with sulphuric acid	41°.5
Melting point	40°.7

Eleven samples of steam lards passed by the Chicago Board of Trade gave as a mean the following data:

Refractive index	1.4623
Percentage of iodine absorbed	62.86
Rise of temperature with sulphuric acid	39°.9
Specific gravity at 35°	.9055
Melting point	37°

Thirteen samples of Armour's mixed lards gave the following data:

Refractive index	1.4634
Percentage of iodine absorbed	63.58
Rise of temperature with sulphuric acid	46°.5
Specific gravity at 35°	.9060
Melting point	40°.6

Sixteen samples of Fairbank's mixed lards gave the following data:

Refractive index	1.4651
Percentage of iodine absorbed	85.31
Rise of temperature with sulphuric acid	57.9
Specific gravity at 35°	.9095
Melting point	38°.1

Sixteen samples of purified cotton oil gave the following data:

Refractive index	1.4675
Percentage of iodine absorbed	106.84
Rise of temperature with sulphuric acid	83.7
Specific gravity at 35°	.9145

Making use of these data we reach the following results:

Determined by the rise of temperature with sulphuric acid, Fairbank's lards contained 32.80 per cent. of cotton oil and Armour's 11.39 per cent. Calculating from their respective specific gravities, Fairbank's lard contained 45.65 per cent. of cotton oil and Armour's 7.60 per cent. Determined by the iodine absorption alone, Fairbank's lard contained 52.29 per cent. cotton oil and Armour's 3.6 per cent. As determined by the refractive index, Fairbank's lard contained 56.36 per cent. cotton oil and Armour's 25.45 per cent.* The unknown quantity, however, is the effect which the stearines employed had upon the data given. An oleomargarine examined in my laboratory had an iodine absorption of 18 per cent.; a prime lard stearine an iodine absorption of 44 per cent. Thus more than twice as much of the oleo-stearine could be used as of the lard stearine without perceptibly influencing the percentage of iodine absorption. The specific gravity of the lard stearine used was about the same as that of the lard, viz., .905, although a larger number of determinations might show a lower specific gravity.

The refractive index of one sample of oleo-stearine calculated at 25° was 1.4646. Before formulating any rule in regard to the matter I readily admit that a much larger number of samples of stearine should be examined and their refractive indices determined. I think, however, it will appear finally that both the specific gravities of the stearines employed and the refractive in-

* The above [illegible] calculated from the mean data for the sixteen samples of mixed lard and the sixteen samples of purified cotton oil.

dices will be found not to vary greatly from the numbers for pure lard; the specific gravities being somewhat lower. It is seen by the above results that the approximate quantity of cotton oil in lard, as indicated by the refractive index, is much nearer the true proportion for the Fairbank and Armour samples than that given by any of the other methods employed. For instance, let us suppose that the Fairbank samples were composed of 60 per cent. cotton oil, 20 per cent. pure lard and 20 per cent. lard stearine; the quantity of iodine absorbed by such a lard would be as follows:

$$\begin{aligned} 60 \times 107 &= 6420 \\ 20 \times 62 &= 1240 \\ 20 \times 44 &= 880 \\ 100 \text{ parts} &= 8540 \end{aligned}$$

Iodine absorption theoretical 85.40 per cent.

Actual iodine number obtained by analysis 85.31 per cent.

Thus from the above theoretical calculation the amount of cotton oil added was 60 per cent., which as indicated by the refractive index was 56.36 per cent., by the specific gravity 45.65 per cent., and by the rise of temperature with sulphuric acid 32.80 per cent.

In the case of Armour's lard take the following:

	Ingredients. Per cent.		Iodine No.		Total.
Pure lard	70	×	62	=	4340
Cotton oil	20	×	107	=	2140
Oleo-stearine	10	×	18	=	180
	100			=	6660

Theoretical iodine number 66.60 per cent.
Actual iodine number . . 63.58 per cent.

Let us suppose again that the compound was made with prime lard stearine, in which case we have the following computation:

	Ingredients. Per cent.		Iodine No.		Total.
Pure lard	70	×	62	=	4340
Cotton oil	15	×	107	=	1605
Lard stearine	15	×	44	=	660
	100			=	6605

Theoretical iodine number 66.05 per cent.
Actual iodine number . . 63.58 per cent.

From the above computations the value of the refractive index in determining approximately the respective quantities of cotton oil and stearine in mixed lards is apparent. It is true that in individual cases the variations might be very much greater than indicated above, but as an expression of the mean results it appears to me that the refractive index is fully as valuable if not more so than the specific gravity in the quantitative determination of mixed lards.

I propose to push this investigation somewhat further by more extensive examinations of the specific gravities and refractive indices of lard, oleo-stearine, prime steam lard and mixed lards.

The refractive index of pure water at 25° as indicated by the instrument employed (Abbe's large model) was 1.3300 When the index of water at the above temperature is taken at 1.3330, .0030 should be added to the numbers given in the above paper.

RECALCULATIONS OF THE ATOMIC WEIGHTS.

BY F. P. VENABLE.

Within the last five years several attempts have been made by chemists of prominence to recalculate the many atomic weight determinations upon a uniform basis and by uniform methods, and so secure, if possible, a trustworthy table of these most important constants of nature. Our better knowledge of these elements and the increased accuracy of modern methods promised favorably for the success of such an undertaking. That such a revision was called for, a glance at the text books of the time will abundantly show. The greatest variance was shown in the tables of atomic weights given. They seemed to be chosen most arbitrarily. No single authority was recognized, and in many cases it would have been difficult to trace the source of the numbers given. Especially were the differences notable in text-books of different nationalities. Taking two nearly contemporaneous text-books widely used in England, America and Germany—Watts (1878) and Richter (1881)—I find that out of 64 elements 37 per cent. only have the atomic weights the same in both; 22

per cent. differ by from .10 to .25; 20 per cent. differ from .25 to .50; 10 per cent. differ from .50 to 1.00, and 11 per cent. differ by more than 1, the difference in several cases ranging from 25 to 40.

To call such a list a table of constants seems ridiculous, nor does it speak well for chemistry as a science that these, the very foundation stones on which its building is reared, should be so unstable and little trustworthy. Analyses calculated by numbers so different, as in these two tables, must give very different results, one or the other, or perhaps both, of which must be erroneous.

The evil was and is a crying one and demands the best energies of the wisest chemists to rectify.

The general acceptation of the Law of Periodicity has been another potent factor in drawing attention to the need for careful revision, and in many cases re-determination of the atomic weights, Those who have undertaken this revision have met with many serious obstacles which are still very far from being overcome. This, I think, will be seen as we proceed in the discussion of the results obtained.

THE UNIT OR BASIS.

The first essential is the adoption of a unit of calculation or basis, and this has proved one of the great obstacles in the way of uniformity. Two elements suggest themselves as bases for these calculations—hydrogen and oxygen. For fifty years or more the strife has raged as to which of these should be adopted. Hydrogen has been practically adopted and used, but the opposition to it has only slumbered at times and seems rising again in the past few years. Dalton and Gmelin were the advocates of the hydrogen unit in early days, and Wollaston and Berzelius advocated oxygen. Among the late revisers and recalculaters, Becker* refers all the atomic weights to oxygen=16. Clarke† gives tables calculated either for oxygen=16 or hydrogen=1. Sebelien‡ uses the units, hydrogen=1 or oxygen=100, as also

*Constants of Nature, Part IV, Smithsonian Institution, 1880.
†Constants of Nature, Part V, Smithsonian Institution, 1887.
‡Beiträge zur Geschichte der Atomgewichte, 1884.

do Meyer and Seubert.* Ostwald† uses hydrogen = 1, giving to oxygen the value 16. Van der Plaats‡ has selected oxygen = 16 as the basis for his recalculation.

There is a decided predominance of authority in favor of oxygen as the standard and of giving it the value 16, though some would make it appear that this is the same thing as adopting hydrogen = 1.

What points should decide the choice of our unit? They have been ably discussed by Meyer and Seubert,§ but I must confess they seem to me somewhat blinded themselves by the partisanship of which they accuse their opponents, and I cannot agree with them in all of their conclusions.

I would state as the essentials for the unit element:

1st. That it must be one with which the greatest number of the other elements can be directly compared, thus avoiding the multiplication of error.

2d. Its own atomic weight must be reasonably small so as not to make too great the higher atomic weights.

3d. The atomic weights of the other elements gotten by comparison with it should be, as many of them as possible, integers or nearly so, rendering calculation easier. In spite of all the tabular and other aids at the command of the chemist of the present day, calculations with an atomic weight having an awkward fraction cause the loss of much time.

Now, on examining with a view to these requisites the two elements proposed as units, oxygen alone will be found to answer every requirement.

Nearly all of the present atomic weights have been determined by the aid of oxygen.

Few can be directly compared with hydrogen, and this forms the almost insuperable objection to hydrogen as a standard.

Hydrogen has been used for the past half century for the two last reasons cited among the requisites. As it has the smallest atomic weight, all the others would be above unity if it were taken as the unit. Thus fractional atomic weights were avoided and

again a large number of the other atomic weights compared with it are approximately integers. I am confident that this is mainly made use of as a matter of convenience and of custom at the present day, and that no special weight is attached to the coincidences with whole numbers. It did give rise to a visionary sort of hypothesis, first enunciated by the Rev. Mr. Prout in 1815, and hence called Prout's Hypothesis, that all of the atomic weights were multiples of that of hydrogen, and, as an inference to be drawn, that hydrogen was the primal element of which they were made.

This hypothesis has had many valiant defenders and a large number of most determined opponents, and it has called forth work that has been of immense benefit to the science. It is well that this one good thing can be spoken of so many false suppositions and theories. As an hypothesis it is based on a few coincidences which were to be naturally looked for in the light of mathematics and the law of probabilities. One may say that no absolute proof in its favor has ever been advanced, nor does it seem capable of proof at the present day. A cursory examination would reveal proportionately similar coincidences for some of the other elements. I say proportionately for, of course, the smaller the atomic weight the greater the number of its multiples and the greater the probability of coincidences within the limits given.

I cannot believe that Meyer and Seubert* are serious when they state it as a "striking fact that the atomic weights of more than one fourth of all the elements are very nearly multiples of the half atomic, or equivalent weight of oxygen," giving a table to show this, and adding that "such regularities are worthy of note." They can scarcely be worthy of much note, for such regularities or coincidences would be exceedingly probable where we have sixty or seventy elements with atomic weights under 240 and take a small number with thirty or more multiples in the same range. The smaller the number the more numerous will be the multiples and consequently the coincidences. To show this, I have added to the table of Meyer and Seubert two other lists of multiples and "regularities."

* Loc. cit.

TABLE No. 1.

Factor.	Multiples of 7.98=½ O.	Elements.	Differences.	Multiples of 3.52=½ Li.	Elements.	Differences.	Multiples of 6=½ C.	Elements.	Differences.
2	15.96	O . . .	0	7.04	Li . .	0	12.00	C	0
3	23.94	Mg . .	0	10.56	Be . .	+.44	18.00	. . . . .	. . . .
4	31.92	S . . .	+.06	14.08	Ni . .	—.04	24.00	Mg . . .	+.38
5	39.90	Ca . .	+.01	17.60	. . . .	. . . .	30.00	. . . . .	. . . .
6	47.88	Ti . .	+.13	21.12	. . . .	. . . .	36.00	Cl . . .	—.55
7	55.86	Fe . .	+.02	24.64	Mg . .	—.26	42.00	. . . . .	. . . .
8	63.84	Cu . .	—.66	28.16	Si . .	—.10	48.00	Ti . .	+.12
9	71.82	Ge . .	+.50	31.68	S . . .	+.38	54.00	. . . . .	. . . .
10	79.80	Br . .	—.04	35.20	Cl . .	+.25	60.00	Co . . .	—.90
11	87.78	Sr .	—.48	38.72	K . .	+.41	66.00	Zn . . .	—.62
12	95.76	Mo . .	+.14	42.24	. . . .	. . . .	72.00	Ger. . .	+.32
13	103.74	Ru . .	—.24	45.76	. . . .	. . . .	78.00	. . . . .	. . . .
14	111.72	Cd . .	0	49.28	. . . .	. . . .	84.00	. . . . .	. . . .
15	119.70	Sb .	—.10	52.80	Cr . .	—.50	90.00	Zr . . .	+.70
16	127.68	. . . .	. . . .	56.32	Fe . .	—.32	96.00	Mo . . .	—.10
17	135.66	. . . .	. . . .	58.82	Co . .	+.28	102.00	. . . . .	. . . .
18	143.64	. . . .	. . . .	63.36	Cu . .	—.03	108.00	Ag . . .	+.06
19	151.62	. . . .	. . . .	66.88	. . . .	. . . .	114.00	Ir . . .	—.30
20	159.60	. . . .	. . . .	70.40	Ga .	—.50	120.00	Sb . . .	+.29
21	167.58	. . . .	. . . .	73.92	. . . .	. . . .	126.00	Te . . .	—.80
22	175.56	. . . .	. . . .	77.44	. . . .	. . . .	132.00	Cs . . .	+.88
23	183.54	W . .	+.06	80.96	. . . .	. . . .	138.00	La . . .	+.50
24	191.52	. . . .	. . . .	84.48	. . . .	. . . .	144.00	. . . . .	. . . .
25	199.50	Hg . .	+.30	88.00	Sr . .	—.48	150.00	Sm . .	+.20
26	207.48	Bi . .	—.18	91.52	Zr . .	—.92	156.00	. . . . .	. . . .
27	215.46	. . . .	. . . .	95.04	Mo . .	+.86	162.00	. . . . .	. . . .
28	223.44	. . . .	. . . .	98.56	. . . .	. . . .	168.00	. . . . .	. . . .
29	231.42	Th . .	+.54	102.08	. . . .	. . . .	174.00	Yb . . .	—.80
30	239.40	U . . .	+.40	105.60	Pd . .	—.60	180.00	. . . . .	. . . .
31	. . . .	. . . .	. . . .	109.12	. . . .	. . . .	186.00	. . . . .	. . . .
32	. . . .	. . . .	. . . .	112.64	Cd . .	—.56	192.00	. . . . .	. . . .
33	. . . .	. . . .	. . . .	116.16	. . . .	. . . .	198.00	. . . . .	. . . .
34	. . . .	. . . .	. . . .	119.68	Sb . .	+.61	204.00	Tl . . .	+.15

TABLE No. 1.—CONTINUED.

Factor.	Multiples of 7.98=½ O.	Elements.	Differences.	Multiples of 3.52=½ Li.	Elements.	Differences.	Multiples of 6=½ C.	Elements.	Differences.
35				123.20			210.00		
36				126.72	I	+.14	216.00		
37				129.24			222.00		
38				132.76	Cs	+.12	228.00		
39				136.28	Ba	+.76	234.00	Th	—1.00
40				140.80	Ce	+.70	240.00	U	—1.00
41				144.32	Di	+.68			
49				172.48	Yb	+.72			
52				183.14	Ta	—.34			
55				193.70	Ir.	—.59			
56				197.22	Au	—.48			
57				200.74	Hg	—.30			
58				204.26	Tl	—.11			
59				207.78	Bi	+.23			
62				218.34	Ng	+.66			
68				239.46	U	+.54			

I cannot think that these authors really mean this as in any way a plea for oxygen$=15.96$. It must be intended to show the ridiculous nature of the grounds upon which Prout's Hypothesis has been based. Certainly they are correct in their deduction that "to attempt to correct the atomic weights by them (*i. e.*, these regularities) would be just as incorrect as to round them off into whole numbers." With such glaring and persistent exceptions as chlorine, chromium, copper, strontium, gold and others, the hypothesis of Prout must fail to take its place as a law, since no law could be accepted with so large a percentage of exceptions.

It is not necessary to discuss the modifications of Prout's Hypothesis which have been proposed—the half-atom or fourth-atom of hydrogen as unit, etc. Such changes really do away with all meaning to the hypothesis, and the valuable idea which Meyer and Seubert acknowledged may lie concealed in it, is lost.

That the hypothesis is still doing yeoman's service to science is shown by the number of new determinations, within the past two years, of the ratio between hydrogen and oxygen. It is evidence of the struggle to retain the old unit hydrogen, and at the same time secure accuracy for it, by fixing definitely its relation to oxygen, by means of which nearly all comparisons with the other elements must be made. I fear that much of this work would not have been done if it had been made to appear that the atomic weight of hydrogen, and not of oxygen, was aimed at. The atomic weight of hydrogen has only the ordinary interest of that of any of the elements. With the atomic weight of oxygen they all stand or fall, no matter which we choose as our theoretical unit. The new determinations referred to are:

Rayleigh (Chemical News, Vol. 57, p. 73), O=15.912.

Cooke & Richards (Am. Chem. Journal, Vol. 10, p. 81), O=15.953.

Cooke & Richards (Am. Chem. Journal, Vol. 10, p. 91), O=15.869.

Keiser (Am. Chem. Journal, Vol. 10, p. 250), O=15.949.

Scott (Chemical News, Vol. 56, p. 175), O=16.01.

Most of these are below the present assumption of O=15.96, so if these investigators are correct, or more nearly correct than those who have preceded them, the whole table of atomic weights must be again shifted.

It is time, then, that hydrogen were finally discarded as the unit. Oxygen is in every way preferable, and nothing like uniformity will be attained until it is adopted.

With oxygen as the standard, what value shall be assigned it? Four values have been suggested: 1st, O=1; 2d, O=10; 3d, O=16; 4th, O=100.

If O=1 we would have nearly 10 per cent. of the elements represented by decimal fractions and the following partial table would show other inconveniences:

Al	1.694	Cr	3.284
F	1.194	Fe	3.501
Mg	1.500	Co	3.67
Na	1.4408	Cu	3.95
P	1.940	Mn	3.43
S	2.0037	Ni	3.67
		Ti	3.15
Si	1.754	V	3.20

The differences between the elements are too small for speedy recognition or for easy memorizing. O=1 is manifestly too small.

Considering next the fourth suggestion, O=100, we see that just the opposite objection holds good. Over one-fifth of the elements would be represented by numbers exceeding 1000. Few of these could be accurately given as far as the fourth place. For instance, shall gold be represented by the number 1229.4, or 1225.1, or 1234., or 1249., all of which are actual determinations? From long custom we have come to regard a whole number as being correct and the decimals as indicating approximations. It would seem to be best still to hide our imperfections under the decimals.

The only claim that can be adduced in favor of O=10 is that we will then have the atomic weights on the decimal system. This is not true, however, unless the other atomic weights are multiples of ten. A glance at a few of the elements will show that the numbers would be complicated, rather than simplified, by such an unit.

H =6.265	F 11.94
Li =4.39	Na =14.408
Be =5.89	Fe =35.01
B =6.83	Co =36.70
C =7.502	Mn = 34.30
N = 8.779	Cu = 39.50

That is, the eight first elements which in our present tables are approximately whole numbers and in ordinary calculations commonly taken as such (especially in technical analysis, and we must not let theoretical considerations take us out of sight of the practical side of chemistry) are when compared with O=10 bur-

dened with cumbrous fractions and brought inconveniently near to one another. This last consideration is of especial weight when the clearness of the Periodic Law is considered.

There remains, then, only O=16 to be considered. The advantage of this unit may be summed up as follows:

1st. Every atomic weight is above unity and yet not inconveniently large.

2d. The distance between the atomic weights renders them easy to memorize and is more convenient for the illustration of the Periodic Law.

3d. About two-thirds of the atomic weights are either whole numbers or vary from whole numbers by fractions of $\frac{1}{10}$ or less.

4th. The adoption of this as a unit practically means the retention of most of the numbers so long in use. The valuable literature of the past three or four decades need not be rendered less useful and intelligible to the chemists of the present and the future, as it would be by radical changes in these combining numbers.

Some chemists seem to feel an especial repugnance to this unit because it is too arbitrary and unusual, not bearing upon its face the fact that it is the unit, and again because hydrogen is then represented by the number 1.0025. The arbitrariness of it seems unavoidable: as to why they should be troubled by the number given hydrogen is not very apparent. The fraction is an easy one to handle, and may often be neglected. It can make no posble difference in the close calculations of organic chemistry cited by Meyer and Seubert, whether H=1 and O=15.96 or H=1.0025 and O=16. The only thing is to keep the ratio which most exact research reveals as existing between the two. The tendency of the day is toward O=16, and it should be universally adopted. To avoid dangerous assumption, the other atomic weights should be rigidly put down in accord with the most accurate determinations available. This brings us to consider another difficulty and cause of variation.

METHODS OF CALCULATION.

Clarke, Sebelien, and others, who have worked over this problem, have been greatly troubled as to what data should be

used in the recalculations and what rejected. Manifestly, the results of many of the older experimenters can lay no great claims to accuracy. Some, as Clarke says, are "chemically worthless because of constant errors." Many have neglected proper precautions or necessary corrections. And yet there are reasons for retaining much of this, and the retention or rejection is a point of judgment on which chemists may, and do, legitimately differ. An additional sentence of Clarke's reveals the extent of the trouble. "In fact, it is doubtful," he says, "whether any two chemists, working independently, would handle all the data in precisely the same way, or combine them so as to produce exactly the same final results."

The accompanying tables, giving the calculations of Clarke, Meyer and Seubert (the O=16 table was worked out from their O=1), Van der Plaats, and Ostwald,* show the truth of this. The two last agree more nearly than the others, and yet the variations are numerous and sometimes as large as 5 in the case of osmium; 3 in the case of mercury; 1 in the case of uranium, etc. Unanimity in regard to an atomic weight does not always mean that the weight is correct. Paucity of data sometimes limits the chances for variation. Indium, for instance, and gallium and beryllium have been subjected to few investigations.

The mathematical side of the question, whether the method of least squares should be adopted, or by what formulæ the probable errors should be calculated, and by what the results combined, is, of course, of great importance, and the subject of varying views, but cannot be discussed here.

*Ostwald's table is put down under the head of O=16. He gives it as H=1 and also O=16, but the numbers most nearly correspond with the latter.

TABLE No. 2.

	OXYGEN=16.					HYDROGEN=1.	
Symbol.	Clarke.	Meyer & Seubert.	Van der Plaats	Ostwald.	Symbol	Clarke	Meyer & Seubert.
Ag	107.923	107.929	107.93	107.938	Ag	107.675	107.66
Al	27.075	27.107	27.08	27.08	Al	27.009	27.04
As	75.090	75.087	75.0	75.0	As	74.918	74.9
Au	196.606	196.690	196.7	196.7	Au	196.155	196.2
B	10.966	10.927	11.0	11.0	B	10.941	10.9
Ba	137.007	137.202	137.1	137.04	Ba	136.763	136.86
Be	9.106	9.103	9.1	9.10	Be	9.085	9.08
Bi	208.001	208.019	208.0	208.01	Bi	207.523	207.5
Br	79.951	79.954	79.955	79.963	Br	79.768	79.76
C	12.0011	11.999	12.005	12.000	C	11.9736	11.97
Ca	40.082	40.009	40.0	40.00	Ca	39.990	39.91
Cd	112 092	111.979	112.1	112.08	Cd	111.835	111.7
Ce	140.747	141.553	141.5	141.5	Ce	140.424	141.2
Cl	35.451	35.458	35.456	35.453	Cl	35.370	35.37
Co	59.023	58.746	58.8 or 60	59.1	Co	58.887	58.6
Cr	52.127	52.581	52.3	52.3	Cr	52.009	52.45
Cs	132.918	133.032	132.8	132.88	Cs	132.583	132.7
Cu	63 318	63.338	63.33	63.33	Cu	63.173	63.18
Di	144.906	145.362	145	145	Di	144.573	145.0
Er	166.273	166.415	166	166	Er	165.891	166.
F	19.027	19.109	19.0	19.01	F	18.984	19.06
Fe	56 042	56.015	56.0	56.00	Fe	55.913	55.88
Ga	68 963	70.072	70.	69.9	Ga	68.854	69.9
Ge	72.32	=Determ	ination of	Winkler.	Ge	....	....
H	1.0023	1.0024	1.00	1.00	H	1.0000	1.00
Hg	200.171	200.299	200.1	200.4	Hg	199.712	199.8
I	126.848	126.856	126.86	126.864	I	113.398	113.4
In	113.659	113.683	113.7	113.7	In	113.398	113.4
Ir	193.094	192.981	193.0	193.11	Ir	192.651	192.5
K	39.109	39.128	39.144	39.136	K	39.019	39.03
La	138.844	138.846	138.0	138.5	La	138.526	138.5
Li	7.0235	7.028	7.02	7.030	Li	7.007	7.01
Mg	24.014	23.999	24.4	24.38	Mg	23.959	23.94
Mn	54.029	54.937	55.0	55.02	Mn	53.906	54.8
Mo	95.747	96.139	96.0	95.9	Mo	95.527	95.9
N	14.029	14.045	14.05	14.041	N	14.021	14.01
Na	23 051	23.052	23.05	23.058	Na	22.998	22.995
Nb	94.027	93.934	94.0	94.2	Nb	93.812	93.7
Ni	58.062	58.746	58. or 58.8	58.5	Ni	57.928	58.6
O	16.000	16.000	16.000	16.00	O	15.963	15.96
Os	198.951	195.487	195	200	Os	198.494	195.
P	31.029	31.037	30.95	31.03	P	30.958	30.96
Pb	206.946	206.906	206.91	206.911	Pb	206.471	206.39
Pd	105.981	106.465	106.5	106.	Pd	105.737	106.2
Pt	194.867	194.826	194.9	194.83	Pt	194.415	194.34
Rb	85.529	85.413	85.4	85.44	Rb	85.251	85.2
Rh	104.285	104.360	104.	103.05	Rh	104.055	104.1
Ru	104.457	103.759	104.	103.8	Ru	104.217	103.5
S	32.058	32.059	32.06	32.063	S	31.984	31.98
Sb	120.231	119.899	120.0	120.29	Sb	119.955	119.6
Sc	44.081	44.079	44.	44.09	Sc	43.980	43.97
Se	78.978	79.067	79.	79.07	Se	78.797	78.87
Si	28.260	28.07	28.0	28.06	Si	28.195	28.0
Sm	....	....	150	150.2	Sm	....	....
Sn	117.968	117.643	118.1	118.12	Sn	117.698	117.35
Sr	87.575	87.518	87.5	87.52	Sr	87.374	87.3
Ta	182.562	182.455	182.8	182.8	Ta	182.144	182.
Te	128.254	128.019	125	125.2	Te	127.960	127.7
Th	233.951	232.539	233	233	Th	233.414	231.96
Ti	49.961	50.373	48.1	48.12	Ti	49.846	50.25
Tl	204.183	204.209	204.2	204.146	Tl	203.715	203.7
U	239.030	240.399	240	239	U	238.482	239.8
V	51.373	51.228	51.3	51.21	V	51.256	51.1
W	184.032	184.059	184.0	184.0	W	183.610	183.6
Y	90.023	89.824	89.5	89.0	Y	89.816	89.6
Yb	173.158	173.081	173	173.2	Yb	172.761	172
Zn	65.054	65.042	65.3	65.38	Zn	64.9045	64.88
Zr	89.573	90.626	90.5	90.7	Zr	89.367	90.4

Out of 66 elements the revisers agree on 29 to the tenth place of decimals, differing in the hundredth place only. In the remaining 37, or 56 per cent., the differences are more or less great.

The increased interest in these redeterminations of atomic weights, giving fresh data for calculation, and enabling us to throw off some of the burden of faulty determinations, gives promise of an approximately correct table in the near future.

I cannot close without adverting to the speculations of some authors as to the question whether we are to expect these atomic weights to be fixed quantities. In other words,

ARE THE ATOMIC WEIGHTS CONSTANT?

This question Stas proposed to himself before starting upon his classic work on the atomic weights. The conclusion he drew from his experiments was that they were unchangeable. The question has been raised again by Schützenberger* and Butlerow.† Butlerow does not doubt the results obtained by Stas, but suggests that under changed conditions or with different bodies the results might have been otherwise.

Of course, if the atomic weights are not constant, the law of constant proportions is without support and must be given up, and this would necessitate a revolution in chemistry as a science.

These authors suppose the range of variation in the weights to be very slight, yet distinctly to be detected by analysis. The theories of both are supported by analytical data, in which the authors seem to place the utmost confidence. If their results are not accurate, the variability of the atomic weights stands unproven. To show the nature of their experiments, Sebelien quotes from Schützenberger's work his synthesis of water. According as this is carried out with copper oxide, at red heat, or by the lowest possible temperature, or with lead chromate, the relation between the oxygen and hydrogen varies from 7.89 to 7.98. Or again, the atomic weight of iron, determined from the nitrate, he finds to be 54., whilst that from the oxalate is 56. He found also that carbon dioxide prepared by burning pure carbon

*Bull. de la Soc. Chim. de Paris, **39**, 258.

†Bull. de la Soc. Chim. de Paris, **39**, 263. Cited in Zeitschrift für anal. Chemie, **22**, 640, and Sebelien, Geschichte der Atomgewichte, 54.

at a high temperature contained more oxygen than that prepared by means of carbon monoxide from organic bodies.

The generality of chemists will be more apt, I think, to suppose the analytical work of these investigators faulty than to accept their conclusions as to the inconstancy of the atomic weights. Yet the matter is of the utmost importance, and should be decided with as great freedom from preconceived notions as possible. It is a question exceedingly difficult to decide, and will require great nicety and accuracy of work. Many of the most trusted leaders of work and thought in the science will have to concur in testimony derived from their own experiments before any attempt at altering the science to suit the new facts will be made.

We must not say, because we are mentally satisfied with the present theories and dread the trouble which so radical a change would cause, that the supposition is impossible and need not be considered.

Butlerow offers three possible explanations of his own and Schützenberger's observations :

1. The absolute amount of matter has been increased in that the so-called force or energy has been changed into matter.

2. The absolute amount of matter is unchanged, but its weight is increased by means of a temporary increase in the intensity with which the earth attracts matter.

3. The weight of matter is not increased in either way, but the chemical value is changed. The atomic weight of carbon, for instance, may be temporarily changed from 12 to 11.8 and thus the saturation capacity of carbon raised by about $\frac{1}{60}$. The amount of carbonic acid made from the same amount of carbon would thus be increased and would be richer in oxygen.

The first two suppositions would be subversive of Natural Philosophy generally. The last would simply be subversive of Chemistry as now systematized. As Sebelien says, we must give up, under the third supposition, our idea of atoms, for an atom is nothing if not a fixed weight of something. Vogel* has also

*Nature, Vol. 41, p. 42.

come to the conclusion that the atomic weights vary because those gotten by the use of certain compounds differ throughout from those derived from other compounds. By this assumption he also explains the cases in which analyses result in a sum total of over 100 per cent. It seems much more plausible to explain these variations on the ground of errors of analysis, constant errors of method, impurities of materials, and the many other difficulties and obstacles which a chemist meets in such work, than by the radical assumption of an inconstancy in the very constants on which the science is founded and built up.

At any rate, until much more proof is forthcoming, the matter must rest in abeyance.

ABSTRACTS.

APPARATUS AND REAGENTS.

New Filter Stand.—D. B. Dott, J. Soc. Chem. Ind. **7**, 830. The base has the form of a truncated isosceles triangle, broad end outwards, and into the bottom 3 wooden, projecting pegs are inserted; this gives the stand 3 supports only, and makes it perfectly steady. The sliding piece has the same shape as the base, but is not so wide, and has two holes—one for large (the outer one) and the other for small funnels.

Apparatus for Regulating the Pressure when Distilling in a Vacuum.—J. Moschner, Chem. Zeit. **12**, 1243. This consists of two bottles, the first provided with three and the second with two necks. The outside neck of the first is connected with the distillation apparatus, and the outside neck of the second with a filter pump. The inside necks are connected with a bent glass tube, one end of which passes through the neck of the first bottle and the other end reaches nearly to the bottom of the second bottle, which is half filled with water. The middle neck of the first bottle is provided with a glass stop-cock, which is opened according to the pressure required. Just before the end of the operation this is opened entirely, to prevent the water in the second bottle from being drawn into the first. S. C.

Improved Positive Electrode.—Alex. Classen. Ber **21**, 360. The round platinum plate 4.5 cm. in diameter, recommended by the author in his work on electrolysis, has the disadvantage that in the electrolysis of iron solutions bubbles of gas collect beneath it, and when they escape round the edge carry particles of the solution, which are projected upon the watch glass and cause the deposition of a film of ferric oxide, resulting in slight loss. This is especially the case if a warm solution is subjected to electrolysis, or if the current is passed for a considerable length of time. It may be obviated by piercing holes in the anode with a cork borer.

Gas Generating Apparatus. G. S. Johnson. Chem. News, **57**, 213. This consists of a bulb blown upon one end of a long glass tube. The bulb is drawn out below into a small opening and contains the solid to be acted upon by the acid (ferrous sulphide, marble or zinc). The bulb is held upright in a cylinder by passing the tube through the cork, and gas is generated by pushing the tube through the cork until the bulb is immersed in the acid contained in the cylinder.

Desiccators for Drying Substances Sensitive to Light. C. Liebermann. Ber. **21**, 2228. The desiccators are made of brownish yellow glass of the same sort as that used for the windows of photographic dark chambers. Funnels, covers and crystallizing dishes of the same glass are also useful.

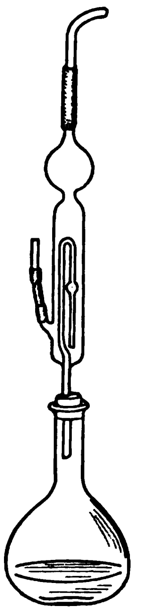
The Polariscope and its Applications to Brewing. J. Heron. J. Soc. Chem. Ind. **7**, 259.

Apparatus for the Rapid Cooling of Crucibles. C. Brunneman. Chem. Zeit. **12**, 1555. This consists of a box made of tinned sheet copper so arranged that water may be made to flow in one end at the bottom and out the other end at the top. In the top of the box are fitted two capsules of the same material just large enough to hold the platinum crucibles that are to be cooled. By passing cold water through the box red hot crucibles may be cooled and ready for weighing in two minutes. S. C.

A Modification of Liebig's Potash Bulb. W. Dittmar. Chem. Zeit. **12**, 1554. The tube connecting the bulbs is extended

about one-third way through the upper bulb and is then bent over so that the end nearly touches the lower side. S. C.

Distilling Apparatus for Nitrogen Determinations.—J. Kjeldahl, Chem. Zeit. Rep. **12,** 238. Two sources of error occur in the author's method for the determination of nitrogen: 1st.—By using a glass condenser the water vapor dissolves some alkali from the glass. 2d.—Particles of alkali are carried over mechanically from the distilling flask. The first of these may be avoided by using a tin condenser. The second is remedied by the apparatus shown in the cut.



Before starting the distillation water is run into the side opening, which is afterward closed by a piece of rubber tubing and a glass rod. This water collects any alkali that may be carried up from the flask and being heated by the water vapor does not retain any ammonia. S. C.

Analytical Apparatus.—G. Neumann J. prak. Chem. **38,** 85. *A New Burette for Gas Analysis.*—A new form of the author's Hydrometer* modified to suit its new applications as shown in Fig. 1. *A* has a capacity of 100 cc. and *B* of 80 cc. divided in $\frac{1}{10}$, the zero mark of the graduation beginning at the three way cock *a*. The tube terminates in a well ground stop-cock *b* with an opening of about 5 mm. Above the cock *a* is a funnel tube with ground neck into which the end of *F* fits. *F* holds 100 cc. and is graduated from the top downwards. The apparatus may be applied to the following purposes:

1. Hydrometer.† The substance which is to give off the hydrogen is put into a 200 cc. flask and treated as already detailed.‡ If the apparatus is to be used as a Bunte's burette also (see below) it is better to add another bulb at *C* as shown by the dotted lines. If the amount of gas given off is less than 100 cc. it is transferred to the upper tube *P* to be measured.

*This Journal **2,** 315, Fig. 3.
†This Journal **1,** 419.
‡This Journal **2,** 315.

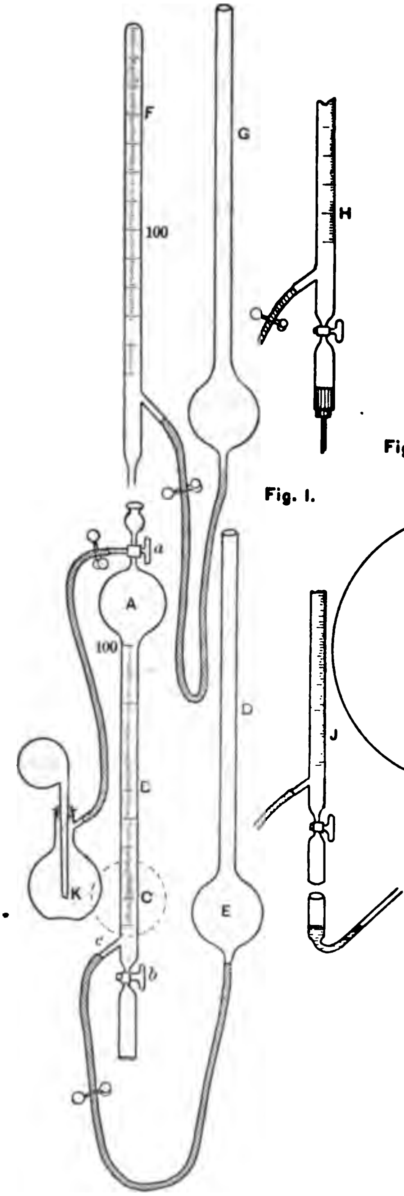


Fig. 1.

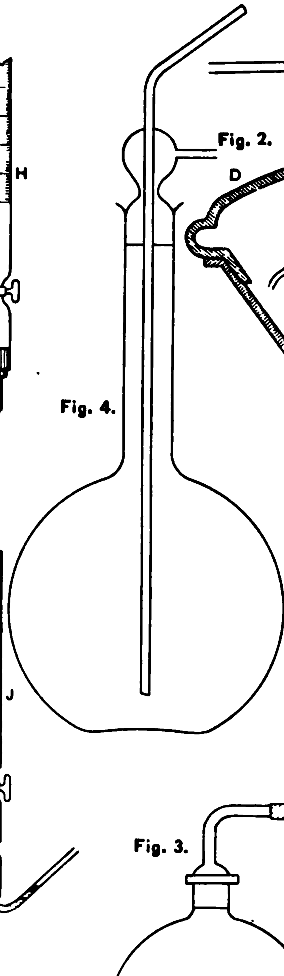


Fig. 4.

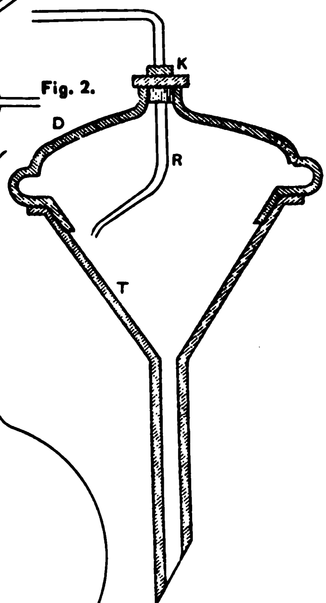


Fig. 2.

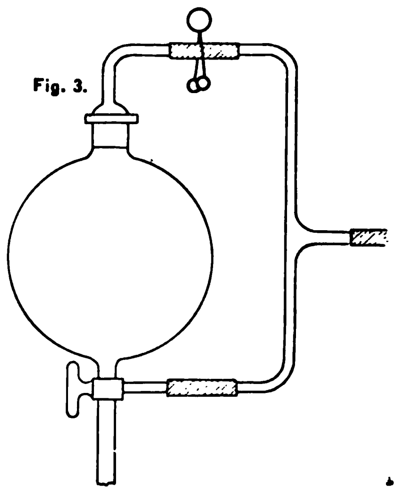


Fig. 3.

2. Bunte's Burette: If the rubber tube connected with *c* is closed with a pinch cock and a cork with capillary tube inserted in the open tube below *b* (see Fig 1 *H*) we have a Bunte's burette which has some advantages over the ordinary form.

3. Zulkowsky's Nitrogen Tube as modified by Schiff: If we open *b* and connect the lower end of the tube by means of rubber tubing with a bent tube containing mercury as shown at *J* Fig. 1 the apparatus can be connected directly with the combustion tube and used in place of Schiff's apparatus.

4. Lunge's Nitrometer: If *b* is closed the apparatus may be used instead of a nitrometer. As a decomposing vessel the author uses a 150 cc. tubulated flask *K* into the neck of which a 100 cc. retort is fastened. The neck of this retort must not be too narrow. The substance to be analyzed is put into *K* and the decomposing liquid in *L* and by inclining the flask the liquid flows into *K*.

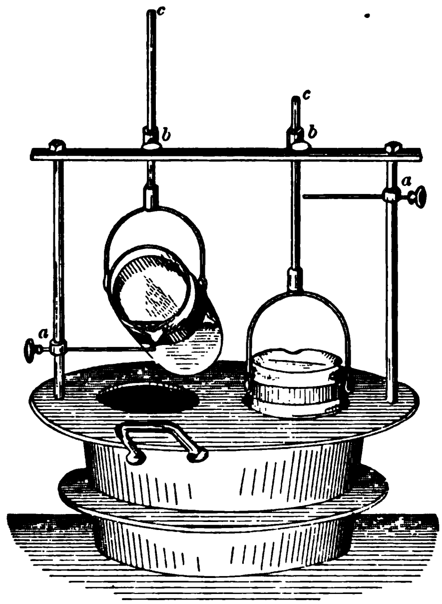
A Convenient Filtering Arrangement is shown in Fig. 2 which needs little explanation. The neck of the funnel is passed through the cork of a flask connected with a pump and the liquid to be filtered is drawn in through the tube *R*. By rotating the funnel and raising or lowering the tube, the stream of liquid can be made to fall upon any desired spot.

A Dropping Funnel with Three Way Cock is shown in Fig. 3. This apparatus is especially convenient when distillation is to be conducted in a current of gas. The lower cock is a three way one and when turned in one position gas passes through the tube of the funnel and on into the flask in which the distillation is being conducted; in the second position the gas stream is cut off and the liquid contained in the funnel passes into the flask.

Fig 4 shows a receiver for use in the same operation, the upper end of the long tube being connected with the condenser tube, and the short side tube the exit which may be connected with an aspirator if desired.

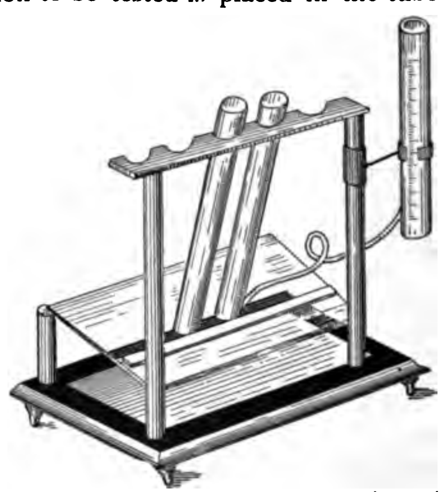
The author uses this funnel tube and receiver for the determination of acetic acid in crude sodium acetate, the acid being distilled off in a current of air after adding phosphoric acid.

A New Oil Bath.—Chem. Zeit. **12**, 1243. The apparatus overcomes the difficulties of removing hot beakers from the bath with the hands and of removing the oil adhering to them.



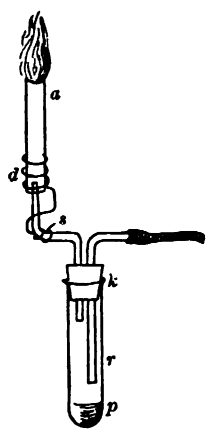
As shown in the cut, the beaker is raised by the rod *c* and inclined by the rod *a* so the oil may drain off. The bath is made of cast-iron, enameled on the inside. The small sizes can be combined with Hoffman's water-bath. S. C.

An Apparatus for Colorimetric Determinations.—A. W. Stokes, Chem. Zeit. Rep. **12**, 174. On the bottom of the stand is a plate of opaque glass, and a little above this is a plate of ordinary transparent glass placed in an inclined position to serve as a rest for the test glasses. The solution to be tested is placed in the tube on the right. The other tube is connected with the graduate by a piece of rubber tubing. A measured quantity of the standard solution is put into the graduate and diluted up to a certain point. This solution is then allowed to run into the other tube, which contains an indicator in solution, until it acquires the same tint as the solution to be tested. This may be done by raising or lowering



the graduate or by attaching a pinch-cock to the rubber tubing. The colors may be compared better by wrapping a sheet of black paper around the tube and allowing the light to come through the bottom. S. C.

Spectroscopic Notes.—H. W. Vogel, Ber. **21**, 2029. *A Convenient Arrangement for Volatalizing Chlorides, etc.*, is shown in the cut. The chloride is placed in the test tube, and a current of coal gas is passed through and lighted at *a*. The coal gas flame is made non-luminous by raising or lowering the upper tube until the correct proportions of gas and air pass through the upper tube.



Spectroscopic Detection of Chromium.—The absorption spectrum gives an excellent means for the detection of chromium salts, but is not so satisfactory for chromates. In this case the chromic acid should be converted into oxychloride (CrO_2Cl_2), which is then volatilized in the apparatus described above. For this purpose the substance is mixed with a little common salt and heated in the test tube until the materials frit together. The moisture is then wiped from the walls of the test tube with a bit of filter paper; after cooling, ½ cc. of sulphuric acid is poured in, the flame lighted, and the test-tube gently heated. If chromium is present, the flame is colored violet red (not unlike the potassium flame) and gives bright lines, of which the most conspicuous are in the author's apparatus (which shows K_a at —23, Li_a at —18, Na at 0, Tl at +18.25 and Sr_a at +57) at —12.5, —4.5, +10 and +24. As little as one milligram chromic acid may be detected by this method. $CuCl_2$, $PbCl_2$, etc., do not interfere since they volatilize at a much higher temperature, and NO_2, HCl and HNO_3 are without influence.

Examination of Thick Layers of Liquid by the Spectroscope.—The amount of light absorbed by a solution increases with the increase of substance dissolved and with the thickness of the absorbing layer. With many substances the thickness of the absorbing layer can be increased until sufficient absorption has been obtained to distinguish the absorption bands. In former

publications the author has proposed to examine thick layers of solutions by placing the liquid in a vertical test tube with the spectroscope above and a mirror to reflect the light below. This arrangement has the disadvantage that the convex bottom of the tube acts as a lens, the rays being drawn together into a focus and then reflected by the walls of the tube, so that only a part of the light reaches the spectroscope. This difficulty may be obviated by using two tubes, one of which fits so snugly into the other that it will remain in any position in which it is placed. A little water is poured into the outer tube, and this forms a plano-convex lens. The inner tube containing the solution is now raised and lowered until the most light is obtained. This arrangement is much superior to that formerly recommended by the author.

Dropping Apparatus.—A Gerngross, Ding. poly. Jour. **270,** 186. The apparatus consists of a tapered glass tube small end downwards; into the smaller end fit dropping tubes of different size. The upper end is fitted with a cork bored with two holes; through one of these passes a siphon dipping into the liquid, placed in a vessel at a higher level, and through the other a short tube communicating with the outer air. The apparatus is set in motion by sucking on the short tube [which is then closed by a rubber tube and clamp, probably, although the author does not say so].

Simple Apparatus for Determination of the Molecular Weight by Raoult's Method.*—Auwers (Ber. **21,** 701) and Holleman (Ib. 860) have described apparatus for making the necessary determinations, of which the apparatus described by Holleman is decidedly the simpler in form, and will be here described. It consists of a test tube provided with a sensitive thermometer, divided in $\frac{1}{100}$ and a stirring rod, bent into a ring at the lower end at right angles to the rest of the rod and surrounding the thermometer. As the cooling vessel, a beaker containing ice water is used, glacial acetic acid being the solvent. The acid (30–40 gms.) containing the proper quantity of substance in solution is poured into the test tube, and this is dipped into the ice water until the temperature falls to about 0.5° below the solidi-

*See also this Journal II, 349.

fying point. The tube is now taken out of the ice water and a small crystal of acetic acid dropped in. This causes solidification, and the temperature immediately rises slightly and is read off with a microscope. The determination should be repeated several times with the same sample and the mean of the determinations taken.

Draught Arrangement for Water Baths.—C. Winkler, Ber. **21**, 3563. Each water bath is placed close to a horizontal slit in the wall communicating with the flue. During the evaporation the bath and contents are covered with a sheet of glass let down over it just above the slit, and supported at the sides by pieces projecting from the wall.

INORGANIC ANALYSIS.

The Estimation of Nitrates in Natural Waters. — Samuel C. Hooker, Ber. **21**, 3302. The author finds that the reaction of carbazol with oxidizing agents first noticed by Graebe and Glaser (Ann. Chem. Pharm. **163**, 347) may be made use of for the detection and estimation of nitrates in natural waters. When a water containing as little as one two-millionth part of nitric acid is treated with a sulphuric acid solution of carbazol a visible green color is produced. Within certain limits the intensity of the color produced is proportioned to the amount of nitrates present.

A measured amount of water, 2 cc. or less, according to circumstances, is mixed with 4 cc. concentrated sulphuric acid and, after cooling, a small quantity of sulphuric acid containing dissolved carbazol added. The color obtained is compared with that from a solution containing a known quantity of nitrate.

Separation of Tin from Antimony.—Alex. Classen, Ber. **21**, 2897. The author has already shown (Ber. **18**, 1108) that antimony may be thrown down by the electrolysis of the solution of the mixed sulphides in sodium sulphide, leaving the tin in solution. After the separation of the antimony, the solution containing the tin may be treated with sulphuric acid and the tin thrown down as sulphide. The sulphide is treated with hydrogen

dioxide in ammoniacal solution until the sulphur is all oxidized and the stannic acid is pure white. Or, the sulphosalt of tin is treated immediately with hydrogen dioxide until the solution becomes colorless, the acid added to separate the stannic acid, neutralized with ammonia, and a little more hydrogen dioxide added. By whichever method obtained, the solution is heated to decompose excess of hydrogen dioxide, and the stannic acid allowed to settle and filtered off. The precipitate is spirited from the filter with a solution of oxalic acid, the part remaining adhering to the filter washed out with hot oxalic acid solution and the stannic acid brought into complete solution by warming. If a small residue of sulphur is left, this must be separated by filtration. The clear solution is run into the platinum dish, a saturated solution of acid ammonium oxalate added (at least 50 cc.), and the solution subjected to electrolysis with a current giving 8–10 cc. gas per minute. The following results were obtained :

CALCULATED.		FOUND.	
Sb.	*Sn.*	*Sb.*	*Sn.*
71.49	32.48	71.57	32.29
——	——	71.55	32.26
——	——	71.48	32.21
——	——	71.55	31.28
——	——	71.50	32.28

The tin is thrown down as a brilliant white coating, which adheres well to the dish.

Separation of Copper from Lead.—Alex. Classen, Ber. **21**, 368. The separation is easily effected and is accurate when conducted as follows : As positive electrode a platinum dish answers and as negative a platinum plate 4.5 cm. in diameter. To the solution (about 120 cc.) 30 to 40 cc. nitric acid sp. gr. 1.2 are added and the solution subjected to electrolysis with a current giving .1 cc. oxyhydrogen gas at first which is increased to a current yielding 3 cc. gas after a tolerably thick layer of lead dioxide has deposited upon the dish. When the deposition is supposed to be at an end 10 cc. water is added and the current allowed to pass for an hour longer. The oxide is then washed with water without interrupting the current, then with alcohol and finally dried at 100° to a constant weight. The following figures show the exactness to be expected :

CALCULATED.		FOUND.	
Pb.	*Cu.*	*Pb.*	*Cu.*
62.51	25.39	62.50	25.30
——	——	62.58	25.50
——	——	62.60	25.35
——	——	62.59	25.30

Estimation of Copper in Presence of Arsenic.—Alex. Classen, Ber. **21,** 366. The separation of copper from arsenic by the electrolytic method is satisfactory if not more than 0.2 per cent. of arsenic be present ; but if the arsenic exceeds this amount the copper is always more or less dark colored at the end of the operation and contains arsenic. It has been proposed to heat this arsenical copper in the dish after deposition to volatilize the arsenic, to dissolve the copper oxide resulting, and subject the resulting solution to electrolysis as before. This method may answer where the amount of arsenic is small, but will not do with solutions rich in arsenic. In this case the separation of the copper from a nitric acid solution is not quantitative, and the deposit after heating is insoluble in nitric acid. The remedy is found in treating the ore or substance to be analyzed with a saturated solution of bromine in hydrochloric acid, whereby the arsenic is volatilized. The solution is evaporated to dryness, dissolved in sulphuric acid, evaporated to drive off the bromine, and after dilution subjected to electrolysis [see also E. D. Campbell, this Journal II, 370].

Estimation of Phosphoric Acid.—G. Linossier, Chem. Cent. **59,** 1442. The nitric acid solution of the phosphate, which must contain neither sulphuric nor hydrochloric acid is heated nearly to boiling and an excess of bismuth nitrate added. The separated bismuth phosphate is washed by decantation and decomposed with sulphuretted hydrogcn. The filtrate containing the phosphoric acid and excess of sulphuretted hydrogen is then titrated with decinormal caustic soda with Poirriers Orange 3 as indicator, to which monosodium phosphate is neutral. This indicator is preferable to phenolphtalein or other indicator to which disodium phosphate is neutral.

Estimation of Chlorine.—G. Linossier Chem. Cent. **59,** 1441. The chlorine is precipitated by means of silver or mercurous nitrate, the precipitate washed, decomposed with sulphuretted

hydrogen, and the solution titrated after filtration. This method is especially applicable to the determination of chlorine in urine.

Absorption of Cyanogen by Aniline.—Jacquemin (C. R. **100**, 1005) proposes the use of aniline as an absorbent for cyanogen in gas analysis. According to Morris Loeb (J. Chem. Soc. **7**, 812) when aniline acts upon cyanogen complete absorption takes place, but on passing a current of carbon dioxide through the aniline a part of the CO_2 is absorbed and hydrocyanic acid is given off. As carbon monoxide is also absorbed by aniline the method cannot be of value. Hofmann distinctly states (Annalen **66**, 129) that hydrocyanic acid is always formed in the preparation of cyananiline.

Electrolytic Determination of Copper. Ber. **21**, [illegible]. 2 to 3 gms. potassium or ammonium nitrate is added to the copper solution, which is then made alkaline by the addition of 10 cc. ammonia for every 100 cc. of solution, containing about 0.1 to 0.3 gms. copper. The solution is submitted to electrolysis and the copper precipitates as a firmly adherent film; without the addition of potassium or ammonium nitrate it is thrown down as a non-adherent, granular precipitate.

Polarization of Tartrate Solutions. J. H. Long (Am. J. Sci. **136**, 351). A solution of Rochelle salt was prepared (20 g. in 100 cc.) and the rotatory power determined; to this solution varying amounts of different salts were added and the effect upon the rotatory power noted. For the detailed results we must refer to the original paper. The greatest observed effect was produced by a solution of thallium sulphate of which 5 g. caused a deviation from the normal of [illegible] 3.43. The sodium and lithium compounds also cause a decrease in the rotation, while potassium and ammonium compounds cause an increase, and these peculiarities may sometimes be applied in the quantitative analysis of salt mixtures. [See also this Journal II, 42 and 421.]

Analytical Examination of Water for Technical Purposes. A. H. Allen (J. Soc. Chem. Ind. **7**, 795). A discussion concerning Clark's soap test and collateral subjects of which it is impossible to give a satisfactory abstract. The original paper should be read in full.

Technical Analysis of Crude Sulphide of Sodium.—B. Sitlek, Chem. Zeit. Rep. **12,** 174. 10 gms. of the material to be examined is dissolved in water, diluted to ½ liter, and filtered. 50 cc. (=1 gm. substance) are titrated with normal acid using plenolphtalein for the indicator. This solution is boiled each time to expel the sulphuretted hydrogen. The excess of acid is then titrated back with normal soda solution. If the sulphide contains more than 1% of thiosulphate the end of the reaction is indistinct. In analyses of the crude sulphide it is necessary to determine the amount of sulphate present also. This is done as follows: 7.1 gms. of the sulphide are dissolved in 50 cc. of water and zinc chloride solution added until neutral and then diluted to 250 cc. The excess of zinc is precipitated in 100 cc. of this solution and is titrated in the usual manner with $\frac{n}{10}$ solutions of barium chloride and potassium chromate. S. C.

Determination of Nitrogen in Nitrates by Kjeldahl's Method.—Otto Foerster, Chem. Zeit. **12,** 1555. The author dissolves 0.5 gms. of the nitrate in 15 cc. phenolsulphonic acid (5% phenol) and then adds from 1 to 2 grams sodium hyposulphite. The flask must be cooled during these operations or the results will be a little low. The remaining part of the analysis is done in the usual manner. S. C.

Determination of CO in the Atmosphere.—C. de la Harpe and Frederick Reverdin, Chem. Zeit. **12,** 1726. The air is filtered through cotton or mineral wool and passed over pure iodic acid heated up to 150° and then into a starch solution. The CO is converted into CO_2 and iodine is liberated which gives the characteristic reaction with the starch solution. S. C.

Separation of Aluminum and Zirconium.—Am. Chem. J. Vol. 11, No. 1, Jan. 1889. Mr. I. Thomas Davis, Jr., separates zirconium in presence of large excess of aluminum by adding to the hydrochloric acid solution of the metals sodium carbonate in slight excess, so as to give a slight precipitate which is very carefully redissolved in very dilute hydrochloric acid. A solution of sodium iodate is added in excess and the whole heated for fifteen minutes, and allowed to stand for twelve hours. The precipitate of zirconium is filtered out and washed with boiling water to

remove traces of any aluminum salts, dissolved in hydrochloric acid and finally precipitated with ammonia, ignited and weighed. The results are accurate. C. W. M.

Volumetric Determination of Antimonic Acid.—G. O. Knorre, Ztschr. angew. Chem. 1888, 155. The method of Weil* (addition of stannous chloride to the boiling solution containing a known amount of cupric chloride until the color disappears) gives results which are not so sharp as is desirable, but the method is a very rapid one. The method of Weller† (reducing $SbCl_5$ to $SbCl_3$ by potassium iodide and distilling off and titrating the iodine set free with sodium thiosulphate) gives very satisfactory results. From 50 cc. of a solution containing 0.0965 Sb_2O_5 the author obtained 0.0966 and 0.0968 grams. Herroun's method‡ depends upon the same reaction, but the iodine is not distilled off. This method does not give accurate results. According to Herroun the alloy is to be dissolved in hydrochloric acid with addition of potassium chlorate and the solution boiled until all smell of chlorine has disappeared; potassium iodide is now added in slight excess and the solution titrated with thiosulphate; but the author finds that after the solution has been decolorized by the thiosulphate more iodine is separated if the solution is allowed to stand a short time, showing that the reduction of $SbCl_5$ to $SbCl_3$ is complete only when the solution is hot and the iodine distilled off. If an excess of thiosulphate is added and the excess determined with iodine and starch sharp results are still not obtained, the results being generally too low. The author also attempted to estimate the antimony by adding potassium iodide, distilling off the iodine and titrating the residual $SbCl_3$ with iodine solution, but the results obtained were too low.

The method of Fleischer§ also gives good results when carried out as follows: To the solution in acid, sodium sulphite solution is added in small portions and the liquid boiled until all excess of SO_2 is driven off; a few drops phenolphtalein solution is added and then caustic potash solution to redness, then tartaric acid solution

* Ztschr. anal. Chem. **17**, 438.
† Ann. Chem. **213**, 364; Ztschr. anal. Chem. **22**, 253.
‡ Chem. News, **45**, 101.
§ Lehrbuch der Titrirmethode, 2d Auflage, 306.

until colorless, the solution supersaturated with sodium bicarbonate and finally titrated with iodine solution.

Volumetric Determination of Lead by Means of Potassium Ferrocyanide.—Yoon. Chem. Zeit. Rep. **13,** 16. This method may be used for the determination of lead in the presence of tin. The metals must be in an acetic acid solution containing no free nitric acid. Two standard solutions are required: 1st, a lead solution made by dissolving 15.987 gms. crystalized lead nitrate in one liter of water (1 cc. = 1 gm. lead). 2nd, a solution of potassium ferrocyanide made by dissolving 10.201 gms. of the crystallized salt in one liter of water. A solution of ferric chloride (1 gm. in 250 cc. water) is used as an indicator. The potassium-ferrocyanide solution is standardized by titrating against 10 cc. of the lead solution. The end of the reaction is shown by the appearance of a blue color when a drop of the solution is brought in contact with a drop of the ferric chloride solution upon a porcelain plate. In case the end mark is passed, the excess of the cyanide solution may be titrated back by the standard lead solution.

In analysis, 0.5—1 gm. of the alloy of lead and tin is dissolved in warm dilute nitric acid, and the solution diluted to 30–40 cc. and neutralized with caustic soda solution until a precipitate begins to form. This precipitate is then dissolved in a slight excess of acetic acid, and the solution diluted to 100 cc. 10–20 cc. of this solution is taken for analysis without filtering off the stannic acid and titrated with the potassium ferrocyanide solution as before. This method is rapid, and gives accurate results when the solution contains not less than ½ % of lead. S. C.

Note on a New Test for Iron.*—Bertram Blount, Chem. News **58,** 195. The use of cobalt as an indicator seems needless since the ferric chloride, formed by the addition of strong HCl, gives its characteristic yellow color. The green color, obtained when the blue cobalt and yellow ferric solutions are mixed, may be obtained by other yellows, as platinic chloride or potassium bichromate, as well. A. H. W.

Determination of Dissolved Oxygen in Water.—L. W.

*See this Journal I, 312; II, 4, 169.

Winkler, Ber. **21**, 2843. If a manganous salt free from iron is added to water containing oxygen, and afterwards sodium hydroxide, the dissolved oxygen combines with the precipitated manganous hydroxide, forming higher oxides. If potassium iodide and hydrochloric acid are now added to acid reaction an equivalent amount of iodine is set free and may be determined by means of sodium thiosulphate.

The manganous chloride used must be free from iron and be about twice normal. The sodium hydroxide should be about 8 times normal, free from carbonate and nitrite; to this about 10 g KI for every 100 cc. is added.

The determination is made in bottles with well ground stoppers, the contents of which must be exactly determined. The bottle is filled with the water to be examined, which should be allowed to flow into it and overflow from the top if possible. About 1 cc. of the sodium hydroxide solution is then run into the bottom of the bottle from a long stemmed pipette and immediately afterwards an equal amount of manganese solution. The stopper is inserted at once, the bottle shaken and the precipitate allowed to subside. The stopper is now removed and about 5 cc. fuming hydrochloric acid added. The contents of the flask are then mixed and the iodine determined in the ordinary way by means of $\frac{n}{100}$ thiosulphate solution, each cc. of which corresponds to 0.05525 cc. oxygen at 0° and 760 mm. If the water contains much carbon dioxide larger quantities of sodium hydroxide and manganous chloride must be used, since manganous carbonate is not oxidized by dissolved oxygen. The author made determinations of the oxygen contained in distilled water saturated with air and obtained in 1000 cc. [illegible] and [illegible] cc. oxygen. By a very ingenious method of his own device, which is fully described in the original paper, he then drove off the air from the same water and determined the amount of oxygen by Bunsen's gasometric method. The results obtained were [illegible] and [illegible] cc. in 1000 cc. of water.

In case nitrites are present in the water an error is introduced and the determination must then be carried out somewhat differently. Instead of adding the potassium iodide with the sodium hydroxide it is added after the addition of the acid. In this way

a small quantity of manganic chloride is produced, which oxidizes the nitrite to nitrate completely before the addition of the potassium iodide, and this has no effect upon the determination. But the result obtained by this method is then too low, and a correction is needed. This is obtained as follows: A solution containing a small amount of manganic chloride is first made by adding to half a liter of distilled water 1 cc. of sodium hydroxide solution (free from KI) and 5–10 drops of the manganous chloride solution; then as much HCl as is needed to dissolve the precipitate (it is better to add a few grams of crystallized manganous chloride, as this helps to dissolve the manganic oxide) is added. Of this manganic chloride solution two portions of 100 cc. each are taken. To the first 100 cc. of distilled water is added, and to the second 100 cc. of the water under examination. Potassium iodide is then added to both samples, and they are titrated with thiosulphate. The difference obtained is the correction to be added in order to give the true result.

The author finds that the amount of oxygen contained in the air from water depends upon the temperature at which saturation takes place, the percentage of oxygen increasing as the temperature falls. For example:

At 0°	35.1 per cent. O
At 10°	34.8 " "
At 20°	34.3 " "
At 25°	33.7 " "

Determination of Phosphoric Acid.—A Grete. Ber. **21**, 2762. The solutions used are as follows:

1. *Molybdic Acid.*—1 kilo ammonium Molybdate is dissolved in ammoniacal water and the solution mixed with a gelatine solution, made by dissolving 100 gms. gelatine in water containing a little nitric acid and boiling for some time. The mixed solution is poured into about 3 liters of nitric acid, sp. gr. 1.2, and then diluted to 10 liters and allowed to stand several days. A flocculent precipitate separates; after removing this the solution keeps perfectly well.

2. *Ammonium Nitrate Solution* containing 75 per cent of the salt and about 40 gms. sodium sulphate per liter.

3. *Glue Solution.*—100 gms. gelatine are dissolved in boiling

water to which about 50 cc. nitric acid has been added and made up to a liter. A few cc. of this solution are sufficient to precipitate 0.1 gm. P_2O_5. If the gelatine solution contains phosphoric acid, as often happens, this must first be removed.

If the above molybdate solution be added to a solution of phosphoric acid containing some gelatine a cheesy precipitate consisting of molybdic acid, phosphoric acid and varying amounts of gelatine is formed; the phosphoric acid solution should first be made nearly neutral. The analysis is conducted by adding to the nearly neutral solution of phosphoric acid a solution of methyl orange and then an equal bulk of the ammonium nitrate solution, and 1 cc. of gelatine solution and heating to 80 to 90°. The molybdic acid solution is then added with constant shaking. The precipitate becomes more and more cheesy and of a deeper yellow color as the molybdic acid solution is added. The molybdic acid solution is run in until a precipitate is no longer produced; this point is easily ascertained since the ready formed precipitate by combination with the methyl orange has taken on a yellow color while the newly forming precipitate is white. From time to time during the titration more gelatine solution is added. If more than enough molybdic solution has been used by mistake a few cc. of phosphoric acid solution of known strength may be added and the titration finished as before. Ammonium nitrate causes too low results while sodium sulphate makes them too high. Much free acid must not be present, or if present should be driven off; small amounts of chlorides are without action. Results:

	[illegible]	
	[illegible]	[illegible]
Superphosphate	0.260[illegible]	0.260[illegible]
Thomas phosphate	0.17[illegible]	0.179[illegible]
Calcium phosphate	[illegible]	[illegible]
	[illegible]	[illegible]
Somme phosphate	[illegible]	[illegible]
Superphosphate	[illegible]	[illegible]

With a little practice a determination may be finished in 5 to 10 minutes by this method. Since the precipitate requires about a minute to settle perfectly several determinations may be carried on at the same time.

Determination of Arsenic in Arseniuretted Hydrogen.—Jacobsen* has shown that when sulphuretted hydrogen containing arseniuretted hydrogen is passed over solid iodine all the arsenic combines with the iodine leaving the sulphuretted hydrogen perfectly pure. Otto Braun (Ber. **21**, 2546) finds that arsenic may be separated in this way from gases containing it and that the arsenic iodide may be dissolved out of the tube and the arsenic estimated as ammonium magnesium arsenate. Antimony is taken up from gases containing antimoniuretted hydrogen in the same way and may be so determined.

Analysis of German Silver.—F. Oettel, Ztschr. anal. Chem. **27**, 15. The author has found that it is impossible to separate the copper perfectly from zinc and nickel by sulphuretted hydrogen in nitric acid solution ; but that the separation is perfect with one precipitation from a sulphuric acid solution. The operation is carried out as follows: The alloy is dissolved in nitric acid and separated from the tin. To the solution 15–20 drops concentrated sulphuric acid is added for each 5 gm. metal and the lead separated in this way as sulphate. The solution is made up to about 100 cc., 2 cc. concentrated hydrochloric acid added and sulphuretted hydrogen passed through it. When the precipitation is finished, which is known by the rapid settling of the separated copper sulphide, the liquid is heated to boiling for a few minutes, cooled, sulphuretted hydrogen passed through again and the precipitate filtered off and washed, first with very dilute hydrochloric acid containing sulphuretted hydrogen and then with sulphuretted hydrogen water. The copper sulphide is ignited in a Rose crucible in a stream of hydrogen and weighed. When the amount of copper is very small it is best to add a little sulphur, but otherwise it is unnecessary to do so.

The filtrate from the copper sulphide is evaporated to drive off the excess of hydrochloric acid, diluted with water, neutralized exactly with potassium hydroxide, sodium acetate added and sulphuretted hydrogen passed through the cold solution until saturated. After standing several hours the zinc sulphide settles perfectly and the supernatant liquid can be poured off and the zinc sulphide washed with sulphuretted hydrogen water without

*This Journal I, 392.

danger of oxidation. It is likewise weighed as sulphide in Rose's crucible.

The filtrate is now evaporated to drive off sulphuretted hydrogen, and a little bromine water added to oxidize the iron [if any excess of bromine water were added here would not manganese also be precipitated?] and the iron precipitated as basic acetate by the addition of a little ammonia. This precipitate must be purified by a second precipitation.

The filtrate from the iron contains the nickel and cobalt and the manganese. This is made alkaline with ammonia and subjected to electrolysis when the nickel and cobalt separate upon the negative electrode as metal, and the manganese, as dioxide, upon the positive electrode. The nickel and cobalt are washed with water and alcohol, dried and weighed. The manganese dioxide is ignited and weighed as Mn_3O_4. The author has found that precipitation of the nickel as hydroxide followed by ignition in hydrogen gives too high results, because of adhering alkali and silica.

The copper may of course also be determined electrolytically in a sulphuric acid solution.

Determination of Arsenic in Pyrites—H. Fresenius, Ztschr. anal. chem. **27,** 34. When pyrites containing arsenic is fused with a mixture of 2 parts sodium carbonate and 1 part niter and extracted with water a part of the arsenic goes into the solution and the rest remains with the oxide of iron. In an experiment tried by the author 0.129% was found in the solution and 0.013% in the residue.

On the Rapid and Sure Detection of Antimony in Minerals.—Alexander Johnstone, Chem. News, **58,** 296. Add, by means of a small glass tube, a drop of ammonium sulphide to the white coating on the charcoal. If the sublimate is composed of antimonious oxide, then the part touched by the edge of the drop will be converted into the reddish or orange sulphide of antimony.

A. H. W.

Volumetric Determination of Sulphur by Means of Baric Chloride.—C. and J. J. Beringer, Chem. News, **59,** 41. After a series of interesting experiments, the plan adopted was as follows: Such substances as pyrite, the sulphur of which can be converted

into sulphate by nitric acid without separation, are thus oxidized, evaporated, taken up with HCl, evaporated again, diluted with water, and transferred to a large evaporating dish; 10 gms. sodium acetate and 5 cc. acetic acid are added, and the solution is diluted to half a liter and boiled. When the substance is not easily oxidized with nitric acid, it is fused with nitre. The solution before titration must contain no free mineral acid, but 5 or 10 cc. acetic acid and a sufficient amount of sodium acetate to convert any free mineral acid into sodium salts. The solution must be kept boiling while titrating, and the barium chloride solution is run in, less than sufficient being used at first, then 3 or 4cc. of the solution is pipetted off and filtered into a test tube and the quantity of the standard solution still necessary is judged by the precipitate. This method permits a determination of sulphur in any sulphide being made in an hour. A. H. W.

Estimation of Titanium in Natural Silicates.—Philip Holland, Chem. News, **59**, 27. It was first found that TiO_2 was stable when digested with HF and H_2SO_4. Experiments were then made to determine the conditions attending a perfect separation of the dioxide, by boiling the acid solution of the bisulphate fusion. The method of analysis finally adopted by the author was as follows: 2 gms. of the sample were put into a large platinum crucible, dilute H_2SO_4 and a little re-distilled HF then added carefully, until 5 gms. had been taken. The covered crucible was next heated in an air bath, first brought to a temperature of 100°C. when the thermometer was removed and the heat raised until the residue appeared dry. Conc. HCl was then added and heat again applied, when, if there had been enough HF in the first instance, the residue dissolved, with the exception of a little TiO_2, which was caught on a filter, and the filtrate was collected in a half-liter flask, in which it was oxidized by a few drops of chlorine water. The contents of the flask (after the addition of the solution obtained by fusing the small TiO_2 residue with bisulphate) was diluted to the mark and divided into two portions D and E, D (= 1 gm. of sample) was diluted and the Al_2O_3, Fe_2O_3 and TiO_2 were precipitated twice by ammonium acetate. The united filtrate was treated for any Al_2O_3 it contained, which was weighed with the rest of the precipitate. After weighing, it was finely powdered

and fused with bisulphate in a tared platinum crucible. The fusion was extracted at 80° C. with water containing a little vitriol, transferred to a large beaker, the solution made just alkaline with ammonia, reacidified with dilute H_2SO_4 (5cc.=0.8 gm. of SO_3), a saturated solution of SO_2 added, and after large dilution with hot water, it was boiled for twenty minutes. The solution was then filtered, the precipitate was fused afresh and the above operations again performed. In portion E. the iron, precipitated first by ammonium sulphide in the presence of tartrate, was washed, dissolved in HCl, oxidized and the hydroxide precipitated with ammonia. The Al_2O_3 was obtained by difference. The air-bath used consisted of a moderately thick unglazed iron dish of 20 cm. diameter with a cone-shaped sheet iron cover, open at top and bottom, 16 cm. high and 9 cm. diameter at its apex, having projecting slips at the sides for handles. There were stout wire tripods and porcelain triangles for support, and a clamp for a thermometer at the top of the cover. The silica and alkalies were determined from fresh portions of the original sample. The author did not secure satisfactory results with G. Roussel's method, which consists in boiling the acid solution of TiO_2, Al_2O_3, and Fe_2O_3 with a solution of SO_2, sodium sulphate and thiosulphate, thus precipitating Al_2O_3 and TiO_2, washing and then gently heating the solution to drive off sulphur, finally digesting in a sealed tube with hot concentrated HCl, which leaves TiO_2 unattacked. The method of the author gave good results for granite, basalts, etc. A. H. W.

On the Rapid Decomposition of Insoluble, Natural and Artificial Silicates for Qualitative Analysis.—Alexander Johnstone, Chem. News, **58**, 310. A small, thin layer of ammonium fluoride is placed on platinum foil or in a platinum crucible, and a little of the finely powdered silicate covered over the fluoride. The platinum is heated with the mouth blowpipe until the ammoniacal fumes cease, then another pinch of ammonium fluoride is added and this operation is repeated five or six times. The assay is then transferred to a small flask containing 40 per cent HCl, sufficient to cover the bottom to the depth of an inch, when it is evaporated one third of the way down, allowed to cool, filtered off, and the filtrate is examined in the usual way.

A. H. W.

Action of Sulphuretted Hydrogen upon Zinc Sulphate in Neutral or Acid Solutions.—H. Baubigny, Chem. News, **59,** 53.

In a zinc sulphate solution, acidified with sulphuric acid, the sulphide is not precipitated at first when sulphuretted hydrogen is passed, but, after a time, depending upon the amount of free acid present, the shorter the less acid there is, the liquid becomes turbid, and the precipitate gradually forms. With 0.3 gm. of the salt to 100 cc. of solution, it is found necessary to add sulphuric acid to about the extent of thirty times the acid present in the salt, in order that such a solution saturated with sulphuretted hydrogen at 0° may not show action at the ordinary temperature, when kept in a closed vessel. With a rise in temperature the sulphide is formed; at 100°, in order to annul the action of the zinc salt, a weight of free acid 90 or 100 times greater than the acid in the salt is requisite. The weight of free acid decreases with the volume of the water in which the salt is dissolved. So many factors enter into the decomposition of zinc sulphate by hydrogen sulphide, that one cannot properly speak of a limit depending upon the degree of acidity of liquid. A. H.W.

Volumetric Estimation of Antimony and Arsenic.—Ad. Jolles. Ztschr. angew. Chem. 1888, 160.

1. *Antimony,*—The antimony as tartar emetic is oxidized to Sb_2O_5 in an alkaline solution with potassium manganate [for the preparation of this salt see this Journal **1,** 400] until the green color of the latter salt is completely discharged. This necessitates adding the antimony solution, first made up to a definite bulk, to a measured volume of the manganate solution. The manganate solution is made by dissolving 4 to 5 gms. of the salt with 8–10 gms. KOH in water and diluting to a liter. The solution must be carefully shielded from organic material and from carbon dioxide. The change of color is from green to a yellowish brown, the following reaction taking place :

$$2C_4H_4O_6KSbO + 2KOH + 2K_2MnO_4 = Sb_2O_5 + 2C_4H_2O_6K_4 + 2K_2MnO_3 + H_2O$$

The potassium manganite formed decomposes sooner or later, yielding potassium hydroxide and manganese dioxide. The rap-

idity of separation of the manganese dioxide depends upon the concentration of the solution. If it should interfere with the sharpness of the final reaction, all uncertainty can be avoided by dipping out a drop upon porcelain and adding a drop of nitric acid; if manganate is still present this forms permanganate known by its color.

2. *Arsenic.*—The method is similar in every respect to that for antimony except that arsenic trioxide may be used for standardizing if thought desirable.

The author has tested his process upon pure tartar emetic and arsenic trioxide and gives results obtained showing the accuracy of the method; but he does not seem to have tested its working with commercial alloys.

Jolles also finds (Ibid 261) that his method is applicable to antimonic acid if this be first reduced by hydrochloric acid and sodium sulphite and the excess of SO_2 boiled off.

Colorimetric Estimation of Iron in Mineral and River Waters.—A. F. Jolles, Chem. Zeit. Rep. **13,** 8.

The water to be tested must not contain more than 0.05 gm. nor less than 0.0004 gm. of iron in every 100 cc. A standard solution of iron ammonium alum is made by dissolving 0.4306 gm. of the recrystalized salt in water and, after the addition of a little dilute hydrochloric acid, diluting to 1 liter. Each cc. of the solution contains 0.00005 gm. iron or 0.00035 gm. ferric oxide.

100 cc. of the water to be tested is placed in a Nessler glass and 5 cc. of ammonium thiocyanate solution (7.5 gms. to 100 cc.) and 1 cc. dilute hydrochloric acid (1.3) are added.

Into four other Nessler glasses 1, 3, 5 and 7 cc. of the standard iron solution are placed with 5 cc. of the ammonium thiocyanate solution in each and enough distilled water to fill them up to the mark. By comparing the colors the amount of iron is easily determined.

In case ferrous iron is present in the water it may be estimated by determining the amount of ferric iron as above and then oxidizing with a few drops of nitric acid and determining the total iron.

The author shows that nitrates, chlorides and carbonates of the alkali metals have no influence upon the color reaction and

that the nitrites, chlorides, and carbonates of the alkali earths are troublesome only in concentrated solutions which never happens in water for analysis. S. C.

Volumetric Determination of Zinc.—Benedikt and Cantor, Ztschr. angew. Chem. 1888, 236.

Zinc carbonate precipitated in the ordinary way by means of sodium carbonate is washed from the filter into a beaker and titrated with hydrochloric acid, using methyl orange as indicator.

The acid combined with zinc in zinc salts may be determined by adding phenolphtalein and then caustic soda solution until the solution is colored red, then a few additional cc. of the standardized caustic soda solution, boiling and determining the excess of caustic soda by running in hydrochloric acid until the color changes. With zinc salts containing an excess of acid, methyl-orange is first added and then caustic soda to neutralization. The zinc in the neutral salt is then determined with phenolphtalein as above.

Quick Methods of Analysis for Industrial Waters.—L. Vignon, Chem. Zeit. Rep. **13**, 8. It has been found in practice that water containing more than 0.3 gm. of solid ingredients per liter is unfit for most industrial purposes and must be purified by chemical means. This is usually done by precipitating the carbonic acid, which is not combined as neutral carbonates, with lime, and converting all the soluble calcium and magnesium salts into the neutral carbonates with sodium carbonate. The author makes use of standard lime and sodium carbonate solutions to estimate the amount of these reagents necessary to purify the water, using phenolphtalein for an indicator. The strength of the lime solution is determined by $\frac{n}{10}$ sulphuric acid solution. Two cylinders are used in the analysis, having a capacity of 100 cc. and a diameter of 2 or 3 cm. Into one of these (A) 50 cc. of distilled water are poured and 10 drops of an alcoholic phenolphtalein solution are added. Into the other (B) 50 cc. of the water to be examined are poured, 10 drops of the phtalein solution are added and 5 cc. of a neutral calcium chloride solution to convert the magnesium carbonate and alkali salts into chlorides. From 0.2 to 0.5 cc. of the lime solution is added to *A* and then to *B* until the color no longer disappears by shaking. Distilled water is then

added to *A* until the bulk of solution is equal to that in *B* and the lime solution added until the colors correspond. From the difference in the amount of lime solution used in each cylinder the amount of carbonic acid not combined as a neutral carbonate may be easily calculated.

To determine the amount of sodium carbonate necessary to convert the alkali salts into carbonates a solution is made by dissolving 1 gm. of sodium carbonate in a liter of water. 100 cc. of the water to be examined are treated with exactly enough lime solution to convert all the free carbonic acid into a neutral carbonate and the precipitate is filtered off. 50 cc. of the clear solution are taken for analysis and treated with the standard sodium carbonate as described for lime.

This method gives quick and accurate results where the water does not contain too much magnesium as a chloride or a sulphate.

S. C.

Volumetric Determination of Chlorine.—John Tsawoo White, Chem. News, **58**, 229.

Suggested by Prof. Hart's scheme* for the qualitative detection of the haloids. The author first separates bromine from the solution, using potassium permanganate but substituting aluminum sulphate for ferric sulphate. The distillation was performed in a flask of about 100 cc. capacity, with a side tube, while a tube for conducting carbon dioxide penetrated the rubber cork almost to the bottom of the flask. The exit tube was connected by rubber tubing with a bent tube, dipping into a solution of potassium iodide contained in a test tube which is kept cold in a beaker of water. The solution was mixed with 1 gm. potassium permanganate and 5 cc. of dilute H_2SO_4 (equal parts of acid and water) the total volume being 50 cc. This is gently heated in a current of carbon dioxide and the potassium iodide solution absorbs the chlorine. Fifteen minutes' heating may suffice. The table of results is appended, the amount taken being determined by silver solution and the iodine set free titrated by $\frac{n}{10}$ $Na_2S_2O_3$ standardized with $K_2Cr_2O_7$.

The bromine in a mixture may be all collected by liberation by $Al_2(SO_4)_3$ and $KMnO_4$, and the iodine of an iodide is set free from

*Ztschr. anal. Chem. **24**, 182.

mixtures with chlorides and bromides by distillation with ferric chloride, chromic acid, and hydrogen dioxide, and thus the volumetric determination of the haloids in a mixture of their salts is rendered possible. A. H. W.

New Method of Determining Carbonic Acid in Solution.—Leo. Vignon, Chem. News, **58**, 298.

This method rests upon the fact that an aqueous solution of carbonic acid, either free, or combined with the neutral carbonates, rapidly decolorizes the red solution, formed by mixing 50 cc. of lime water and 10 drops of a saturated alcoholic solution of phenolphthalein. 50 cc. of the water under examination are mixed with 10 drops (0.05 cc.) of a saturated alcoholic phenolphthalein solution, and lime water, previously standardized, is then added until the characteristic rose shade is produced. For comparison, 50 cc. of distilled water or of the same water as the sample are taken and 10 drops of the phenolphthalein are added and then lime water until the desired tint is reached. The difference between the lime consumed by the examined water and that which colored the type corresponds to the carbonic acid in the water. A little neutral calcium chloride must be first added to convert into chlorides alkaline salts if they are present, where their acids are capable of forming insoluble salts of magnesium and calcium carbonate. Where there is much carbonic acid in the water, and the liquid becomes opaque on the addition of the lime water, some pure calcium carbonate may be added to the type, or that in the sample may be allowed to settle. A. H. W.

The Volumetric Estimation of Cobalt, in Presence of Nickel, Manganese and Other Metals.—Norman McCulloch, Chem. News, **59**, 51.

Cobalt cyanide is a powerful reducing agent, effecting the complete or partial reduction of many metallic oxides, and the reducing effect of a metallic cyanide on chromic acid, (as represented in the equation

$$6Co(CN)_2 + 24KCN + 2CrO_3 + 3H_2O = 6Co(CN)_6K_3 + Cr_2O_3 + 6KOH$$

being peculiar to the cobalt cyanide, where chromic acid is reduced in cyanide solution we may justly conclude the cobalt compound is present, and by the indirect estimation of the reduced chromate with a standard ferrous salt, we may calcu-

late the amount of cobalt. The author uses the following standard solutions: Standard Normal Bichromate (49.17 grains in 10.000 of water) delivered from a burette graduated to fifths of a division (1 div. = 10 grains = 0.059 grain of cobalt.)

Standard Ferrous Salt. ($FeSO_4NH_42Soh_4 + 6H_2O$) standardized with bichromate in the usual way.

Potassium Cyanide Solution of any convenient strength, the author using a five per cent solution of 95 per cent pure cyanide, 12.5 divisions of the solution is theoretically sufficient for one grain of cobalt. This solution may be standardized by adding it to a hot aqueous solution of 3 grains pure nickel sulphate until the precipitate dissolves, and considering the amount thus used, plus one half more, as equivalent to 1.14 grains of cobalt in the process. A 12 oz. flask, with a two-holed cork, one for a thistle funnel, the other for escape of vapors, will answer, and the end of the funnel must be beneath the surface of the liquid in the flask. The standard bichromate and cyanide solutions in proper quantities having been introduced into the flask, a few drops of ammonia added to neutralize any free acid and the solution boiled to expel air from the flask and the hot solution to be tested then added. A warm concentrated solution of ammonium chloride is further added and the boiling is contlnued for ten minutes longer, best in a hood to carry off poisonous fumes. Nickel sulphate must now be added to throw down soluble cobalt cyanide and decompose potassium nickel cyanide. Then the contents of the flask are poured into excess of a hot aqueous solution of standard ferrous sulphate in a basin, acidified with a few drops of hydrochloric acid and titrated with bichromate in the usual way. The cobalt is calculated by multiplying the difference between the number of divisions of bichromate taken at first and that found at the end by 0.059 grain and correcting this by a slight allowance for the reducing action of the potassium cyanide on the chromate. This method will not give satisfactory results when iron and copper are present, but in the presence of other metals very satisfactory results were obtained. A. H. W.

Detection of Very Small Quantities of Arsenic.—F. A. Flückiger, Chem. Zeit. Rep. **13**, 26. The author finds that a concentrated solution of silver nitrate makes a very delicate test

for arsenic, forming the yellow compound $AsAg_3+3AgNO_3$ with arseniuretted hydrogen. As small an amount as $\frac{1}{1000}$ mg. of As_2O_3 has been detected in this way.

A solution of mercuric chloride when acted upon by arseniuretted hydrogen gives almost as sharp a reaction. When this solution is absorbed by blotting paper and exposed to arseniuretted hydrogen a yellow stain is produced at first which gradually changes to a brown. Zinc and hydrochloric or sulphuric acid are better for producing hydrogen than sodium amalgam, but the zinc must be entirely free from sulphur, arsenic, phosphorus, and antimony, so that when 10 gms. of the metal is dissolved in dilute hydrochloric acid the silver nitrate or mercuric chloride paper remains unchanged after being exposed to the gas for two hours, care being taken not to expose the silver nitrate paper to the light. The mercuric chloride solution is more convenient than the silver nitrate solution, since it is not affected by light. S. C.

IRON AND STEEL.

EDITED BY P. W. SHIMER.

The Determination of Sulphur in Iron.—C. Meineke, Ztschr. angew. Chem., 1888, 376.

The author gives a series of results showing that from 0 to 100 per cent. of the sulphur in iron may remain in the insoluble residue after solution of the iron in dilute HCl as in the evolution methods for the determination of sulphur. By dissolving the iron directly in oxidizing solutions it is difficult to get a pure, white barium sulphate. To take the place of these methods the author proposes the following: 5 gms. of iron and about 50 gms. cupric ammonium chloride are treated in a beaker with 250 cc. hot water. Immediately after this add 10–15 cc. HCl. Keep the solution near the boiling point till all the precipitated copper is dissolved. Solution is usually complete in 15 minutes. The solution is filtered through asbestos and washed with hot water containing at first some HCl. The residue is removed from the funnel into a beaker and a little potassium chlorate is added to it to assist the oxidation of the sulphur and to furnish a base for the sulphuric acid. 5 cc. HNO_3, sp. gr.

1.40, and 10 cc. HCl, sp. gr. 1.19 are added and heated on the sand bath, and finally evaporated to dryness. Redissolve in HCl and evaporate till all HNO_3 is removed; dilute and filter. Precipitate the filtrate with barium chloride in the usual manner.

The reaction of the copper salt on the iron is said to be the following:

$$Fe + FeS + 2CuCl_2 = 2FeCl_2 + CuS + Cu$$
$$CuS + Cu + 2CuCl_2 = 2Cu_2Cl_2 + S$$

Estimation of Titanium and Phosphorus in Iron Ores. E. P. Jennings, Eng. and Min. Jour., **45**, 675.

From 1 to 5 gms. of the finely pulverized ore are dissolved by boiling with strong HCl. The solution, containing all or nearly all the titanic acid, is diluted without previous filtration, nearly neutralized with carbonate of soda and the iron reduced with sulphurous acid; 50 cc. acetic acid is added, and the whole diluted to about 500 cc. and boiled for an hour. The titanic acid is all precipitated, together with some phosphorus, a little iron and the insoluble residue from the first treatment with HCl. The precipitate is filtered and washed with hot water containing 10 per cent. of acetic acid, which prevents titanic acid from running through the filter. The precipitate is dried, ignited and fused with ten times its weight of carbonate of soda; the fused mass is treated with hot water to dissolve out phosphate and silicate of soda. The insoluble titanate of soda and oxide of iron are filtered off, the filtrate being added to the first filtrate for phosphorus determination. The titanate of soda is washed from the filter into a beaker and dissolved in dilute sulphuric acid, nearly neutralized with carbonate of soda, a little sulphurous acid and 25 cc. acetic acid added, the whole largely diluted and boiled. The titanic acid is precipitated pure and should be washed as before with dilute acetic acid.

The phosphorus is determined by oxidizing the solution with nitric acid, precipitating the iron and phosphoric acid with ammonia, filtering, redissolving in hot dilute nitric acid, separating silica by evaporating to dryness, redissolving in nitric acid and precipitating phosphorus with molybdate solution in the usual way.

In case more than two gms. of ore are taken, the precipitate

of ferric hydrate will be too bulky for easy filtration and the following method may be used: The solution is neutralized with ammonia; a few drops of ferric chloride added and the whole boiled. The precipitate of basic ferric acetate will carry down all but slight traces of the phosphorus. The precipitate is dissolved in hot dilute nitric acid, evaporated to dryness, redissolved and precipitated with molybdate as before.

The Electrolytic Method as Applied to Iron.—Edgar F. Smith, Am. Chem. J. **10**, p. 330.

To the iron is added an excess of sodium citrate (.015 gms. Fe to 10 cc. sodium citrate solution; made by dissolving 28 gms. of the air-dried salt in 250 cc. water) and a few drops of citric acid. The solution is electrolyzed in a platinum dish by use of a current liberating 6–15 cc. oxyhydrogen gas per minute. The iron is completely deposited in 4–8 hours and has a compact, regular, and steel-like appearance. It is washed with water, alcohol and ether, then dried and weighed. Several deposits suffered no alteration when exposed for weeks to the air of the balance room. Aluminum and titanium, when present, remain completely in solution. Solutions of titanium alone, when treated just as the iron solutions were, gave no deposit. The author promises an account of experiments in the separation of iron and titanium by electrolysis.

Estimation of Ferric Oxide in Iron Ores.—L. Blum, Ztschr. anal. Chem. **27**, 146.

Abstracted in J. Soc. Chem. Ind., VII, 456.

From four to five gms. substance are dissolved, the SiO_2 separated, and the solution made up to 500 cc. 50 cc. are drawn off, tartaric acid added in sufficient quantity to prevent precipitation on adding NH_4OH, the solution is diluted and ammonium sulphide added. The sulphide of iron is dissolved in dilute HCl, oxidized, and precipitated with ammonia, and weighed as Fe_2O_3. The author finds that by the above method, which is often adopted for separating iron and aluminum in iron ores, too high a result is obtained owing to the precipitation of ferric phosphate with the oxide, the phosphoric acid being present in the ore as magnesium phosphate. To avoid precipitation of the phosphate,

the sulphide of iron is precipitated from the hot very dilute solution, and only half an hour allowed for the sulphide of iron to settle before filtering. In this way precipitation of magnesium phosphate is avoided.

MINERAL ANALYSIS.

EDITED BY JOHN EYERMAN.

Anerlite, a new Thorium Mineral from North Carolina.— Hidden and Mackintosh (Am. J. Sci. XXXVI p. 461) give a description and several analyses of *Anerlite* a thorium phosphate from the Freeman mine in Henderson County N. C. The mineral was observed while mining zircon at this well known locality: color from pale lemon-yellow to deep brown-red; crystallization tetragonal; subtranslucent to opaque; very brittle and easily crumbled; H 2.5 to 3; sp. gr. 4.422 to 4.766. One complete and four partial analyses are given; the one complete analysis afforded:

H_2O } CO_2 }	11.21
SiO_2	7.64
P_2O_5	7.46
ThO_2	70.13
Fe_2O_3	1.38
CaO	0.49
MgO	0.29
Al_2O_3 etc.*	1.10
	99.70

"We have (say the authors, p. 462) considered it possible that this mineral is a mixture of hydrated thorium phosphate with a hydrated thorium silicate, in some respects analogous to the occurrence of zircon in parallel position with xenotime† although there is nothing in the appearance of the mineral to suggest this. * * * * It is more probable, however, that we have here an example of a partial replacement of silica by phosphoric acid, which fact has not yet, to our knowledge been noticed * *, unless we should so regard the small proportion of P_2O_5 (4.17) which Eakins‡ observed in the xanthitane from the same locality."

* Including other oxides with traces of thoria.
† Am. J. Sci. XXXVI, p. 380.
‡ *Ibid* XXXV, p. 418.

Hohmannite and Amarantite.—*New species from Chili.*—Two new iron sulphates from Caracoles, Chili, are described by Freuzel (Min. u. Petrog. Mitth., IX., 1888, pp. 397, 423). Hohmannite, named after its discoverer, is a fibrous mineral, with vitreous lustre, color chestnut brown. H. 3; Sp. gr. 2.24; streak ochre yellow. Composition Fe (FeO) $(SO_4)_2+7\ H_2O$. Insoluble in water.

Amarantite is probably identical with the above. Color orange; sp. gr. 2.11; streak citron yellow; crystallizes in the triclinic system.*

Edisonite, a new form of Titanic Acid.—Hidden (Am. J. Sci. XXXVI, p. 272) describes this fourth form of titanic acid from the Whistdant Gold Mine, Polk Co., N. C., under the name of Edisonite (after the inventor T. A. Edison).

Hardness 6.; sp. gr. 4.26; luster resinous to adamantine; color bronze-yellow to golden-brown; streak yellowish white; insoluble in acid.

ATOMIC WEIGHTS.

EDITED BY F. W. CLARKE.

Tin.—The atomic weight of this metal has been studied by Bongartz and Classen (Berichte **15**, 2900) by five methods.

First.—Pure tin was oxidized by nitric acid to SnO_2. In mean, eleven experiments gave Sn=118.7606, if O=15.96.

Second.—The ammonium stannichloride, $(NH_4)_2SnCl_6$, was electrolyzed, and its contents in metallic tin so determined. Sixteen experiments give a mean value of Sn=118.8093, when Cl=35.37, and N=14.01.

Third.—Ten similar experiments were made upon the electrolysis of K_2SnCl_6. In mean, if K=39.03, Sn=118.7975.

Fourth.—Ten electrolyses of the bromide, $Sn\ Br_4$.

If Br=79.76, Sn=118.7309.

Fifth.—Pure tin was converted into chloride, and then precipitated as sulphide. The amount of sulphur thus taken up was determined by Classen's method as barium sulphate. In eight experiments Sn=118.696.

* Wülfing: ib., p. 401.

The last value is not favorably regarded by the authors, who give us the mean of the other four series the figure

$$Sn = 118.7745.$$

If $O = 16$, they assign to tin the value 119.1.

This is a unit higher than the number hitherto generally accepted.

ORGANIC ANALYSIS.

Composition of the Drying Oils.—K. Hazura Ztschr. angew. Chem. 1888, 312. Hazura has published a number of memoirs in the Monatshefte für Chemie upon this subject and gives a summary of his results in this paper. He finds that Mulder's linoleic acid is not a homogeneous substance but a mixture of three acids. The fatty acids from linseed oil consist of a mixture of 20 per cent. oleic acid, 65 per cent. of a mixture of linoleic and isolinoleic acids ($C_{18}H_{30}O_2$) and 15 per cent. of linolic acid ($C_{18}H_{32}O_2$). Hemp oil contains 70 per cent linolic acid, 15 per cent. linoleic and isolinoleic and 15 per cent. oleic acid; cotton seed oil contains 40 per cent. oleic acid and 60 per cent. linoleic acid.

Estimation of Benzene in Illuminating Gas.—F. P. Treadwell and H. N. Stokes, Ber. **21**, 3131. When illuminating gas is treated with fuming nitric acid to remove benzene the amount absorbed depends upon the time during which the gas is shaken with the acid. Not only benzene but carbon monoxide also is absorbed and converted into carbon dioxide, which remains in solution, while pure oxygen is given off. Fuming nitric acid is not, therefore, a proper reagent for the absorption of benzene if the gas contains carbon monoxide.

The authors find that bromine water absorbs benzene from a gas and is not, therefore, a proper reagent for the separation of gases of the ethylene series from benzene. No accurate method is known for the separation of benzene from the ethlene series and only the "total heavy hydrocarbons" can be determined.

Estimation of Acetone in Methyl Alcohol.—J. Messinger, Ber. **21**, 3366. The action of iodine upon acetone in an alkaline solution is expressed by the following reactions:

$$2CH_3CO.CH_3 + 12KOI = 2CH_3CO.CI_3 + 6KOH + 6KI.$$
$$2CH_3CO.CI_3 + 2KOH = 2CHI_3 + 2KC_2H_3O_2.$$

Any excess of iodine added will remain in solution as potassium hypoiodite and iodide.

$$I_2 + KOH = KOI + KI + H_2O.$$

According to this statement 3 molecules of iodine are needed for every molecule of acetone present. After the action is completed, the solution acidified ($KOI + KI + 2HCl = I_2 + 2KCl + H_2O$), and the free iodine titrated with sodium thiosulphate and the amount found subtracted from the total amount used. For the analysis are needed: I. $\frac{1}{5}$ normal iodine solution. II. $\frac{1}{10}$ thiosulphate solution. III. Hydrochloric acid, sp. gr. 1.025. IV. Caustic potash solution (56 gms. KOH per liter). V. A flask of 250 cc. with well ground stopper. VI. A 1 cc. pipette divided in 10 or 100 parts. VII. Starch solution.

As commercial caustic potash nearly always contains nitrites, 20 cc. is measured out, .1 to .2 gms. potassium iodide added, the solution acidified with the hydrochloric acid and the iodine set free determined with the thiosulphate solution, after having added some of the starch solution. The amount of decinormal thiosulphate used must be subtracted from the amount found necessary in subsequent analyses.

THE ANALYSIS.

20–30 cc. caustic potash solution and 1–15 cc. methyl alcohol are measured into the stoppered flask and well shaken; then a sufficient amount (20–30 cc.) iodine solution is run in, the mixture shaken for ¼ to ½ minute until the solution appears clear. Hydrochloric acid is then added until the solution becomes acid (as much as was added of caustic potash solution,) then starch, then sodium thiosulphate in slight excess, and, finally, iodine solution until the blue color appears.

CALCULATION.

As one molecule acetone (58) requires 3 molecules iodine (762) to form one molecule of iodoform, then

$$752 : 58 = m : y$$

m = iodine found

y = corresponding amount of acetone

$$y = m \frac{58}{762} = 0.07612$$

If n cc. methyl alcohol have been taken, then 100 cc. methyl alcohol contain X gms. acetone.

$$n : m. 0.7612 = 100 : X$$
$$X = \frac{m}{n} . 7.612$$

Since ordinarily n is equal to 1, the weight of acetone in 100 cc. alcohol may be found by multiplying the iodine found by 7.612.

$$X_1 = m. 7612$$

With $\frac{1}{5}$ normal iodine solution 1 cc. contains 0.0245 gms., then for 1 cc.

$$X_{11} = 0.0254 \times 7.612 = 0.1933448 \text{ acetone}$$

X_{11} is equal to the weight of acetone in 100 cc. alcohol which one cc of $\frac{1}{5}$ iodine solution will decompose. If r cc. iodine were used.

$$X = \frac{r. x_{11}}{n}$$

TEST ANALYSES.

The author has obtained the following figures in examining into the accuracy of the process:

CALCULATED.		FOUND. 1	2	3	5	
% Acetone	(a) 2.05	2.04	2.03	2.04	2.03	
"	(b) 1.48	1.45	1.44	1.44	1.44	
"	(c) 1.07	1.00	1.03	1.03	1.07	
"	(d) 0.89	0.88	0.85	0.84	0.89	0.88
"	(e) 0.72	0.70	0.70	0.70		

Additional figures were obtained, but the above are sufficient.

NOTE.—It would seem that the author must be mistaken, either in the reaction or the calculation, for if

$$12I_2 + 24KOH = 12KOI + 12KI + 12H_2O \text{ and } 12KOI + 12KI + 2CH_3COCH_3 = 2CHI_3 + 2KC_2H_3O_2 + 4KOH + 18KI$$

it is perfectly evident that not 3 but 6 molecules of iodine are required to form one molecule of acetone, or that 58 acetone = 1524 iodine. E. H.

Determination of Nitrogen and Phosphoric Acid in Organic Substances.—Otto Lange, Chem. Zeit. **12,** 1587. 10 gms. of the substance to be analyzed are placed in a flask of 300-350 cc. capacity and to this are added 50 cc. conc. sulphuric acid and 0.5–1 gms. of copper sulphate. The contents of the flask are then carefully heated until the organic matter is carbonized, after which the solution is heated to a lively boil. At the end of two or

three hours the reaction is complete, which is recognized by the clear green color of the supernatant liquid. After cooling, the solution is diluted to exactly 500 cc. and filtered. 50 cc. (=1 gm. of the substance) of the filtrate are taken for the phosphoric acid determination. To these are added 100 cc. of Maercker's citric acid solution* and 25 cc. of magnesium mixture. This solution is stirred for 15 minutes, filtered and the precipitate weighed in the usual manner. 50 cc. of the original filtrate are taken for the nitrogen determination, and, after diluting with water, the ammonia is distilled off by the addition of an excess of caustic soda solution and some pieces of zinc. This solution boils very quietly.

The previous separation of the copper with potassium sulphide solution is unnecessary, for the oxyammonium salts of copper are completely decomposed by the caustic soda solution. Instead of using copper sulphate in the first part of the operation, 0.5 gms. of mercury may be used which requires only 1–1¼ hours of heating, but a source of error arises from the formation of mercurammonium compounds. S. C.

Direct Detection of Chloral and Chloroform in Liquids.—C. Schwarz, Chem. Zeit Rep. **12**, 211. The solution to be tested, with the addition of a little resorcin, is heated to boiling with an excess of sodium hydroxide. If chloral hydrate or chloroform be present, even in as small a quantity as $\frac{1}{100}$%, the solution will be colored red.

An equally delicate test is obtained by boiling the solution to be tested with an excess of resorcin and a little sodium hydroxide when the presence of chloral hydrate or chloroform will give a red color which by largely diluting the solution gives a yellowish green fluorescence. These reactions are more delicate than any other, even the isonitrile reaction. S. C.

Detection of Cotton-seed Oil in Olive Oil.—H. Levy, Chem. Zeit. Rep. **12**, 238. The author finds the common methods of little use where the adulteration is less than 25%. He recommends the method of Conroy as the best. 30 drops of nitric acid are carefully mixed with one fluid ounce of the oil to be tested and the mixture is heated in a porcelain dish of at least 10 ozs. capa-

*1500 gms. citric acid are dissolved in 3 liters of water and to this are added 5 liters 24% NH_4OH and 7 liters of water.

city. As soon as the reaction begins the flame is removed and the mixture is stirred with a glass rod until the action ceases. Pure olive oil changes, after standing one or two hours, to a light straw colored hard mass, while cotton seed oil becomes a deep orange color and does not harden. In hot weather the mixture must be cooled in order to bring the required hardness. after some practice the extent of the adulteration may be determined approximately by the coloration of the mixture. S. C.

Iodine Absorption, Molecular Weights, and Melting Points of Certain Fatty Acids. R. Williams, Chem. Zeit. Rep. **12**, 127.

Name of Fat	Iodine Absorption	Molecular Weight	M. P.
Tallow	41.3%	276	119°F
Lard	64.2	296	100
Cotton-seed Oil	115.7	289	96
Olive Oil	90.2	286	81
Linseed Oil	178.5	283	75
Rape seed Oil	105.6	307	71
Castor Oil	93.9	292	
Cocoanut Oil	9.3	201	75
Palm Oil	53.4	263	114
Sperm Oil	88.1	305	56

The iodine absorption is higher in the fats and oils than in the corresponding fatty acids. S. C.

Method for Detecting the Adulteration of Pepper with Olive Husks. M. Gillet, Bull. Soc. Chem. **50**, 173. 1 gm. olive husks treated with 1 cc. of 5 per cent. tincture of iodine and the alcohol allowed to evaporate leaves the husks of a bright yellow color. Pepper treated in the same way becomes brown or maroon colored. By treating mixtures of known composition with tincture of iodine as above and comparing the color with that obtained with the sample under examination it is possible to determine pretty accurately the extent of the adulteration.

Change in Rotatory Power of Grape Sugar Caused by Inactive Substances. R. Pribram, Ber. **21**, 2599.

New Method for Elementary Analysis in the Wet Way. J. Messinger, Ber. **21**, 2910.

ESTIMATION OF CARBON.

The substance is placed in a small flask and warmed with a

mixture of chromic and sulphuric acids, which converts the carbon into carbon dioxide ; this is absorbed in a potash bulb and weighed. Volatile substances cannot be burned completely in this way.

ESTIMATION OF SULPHUR.

The substance is heated in a flask with reflux condenser either with an alkaline permanganate solution, chromic acid recrystallized from nitric acid, or a mixture of potassium dichromate and hydrochloric acid. The sulphur is precipitated as barium sulphate. Some substances cannot be oxidized satisfactorily in this way, as, for example, sulphonal and helianthin.

ESTIMATION OF PHOSPHORUS ARSENIC AND ANTIMONY.

The substance is oxidized with chromic and sulphuric acids in a flask with reflux condenser. The phosphorus is determined by means of ammonium molybdate solution in the usual way. Arsenic is precipitated from the solution with sulphurretted hydrogen ; the sulphide is oxidized with hydrogen dioxide according to Classen's method * and precipitated as ammonium magnesium arsenate.

Antimony is converted into sulphide by adding potassium hydroxide until alkaline, then potassium sulphide, boiling for half an hour and precipitating the metal electrolytically by Classen's method.†

The halogens may be detected qualitatively in substances containing them by oxidizing with chromic and sulphuric acid and leading the vapors given off through potassium iodine solution when iodine is set free. The effort to make this a method for quantitative determination failed, the results obtained being slightly too low.

Determination of Paraffine.—Pawlewski and Filemonowicz, Ber. **21,** 2973. The authors have determined the solubility of paraffine in various liquids and find that 1 part dissolves in 1668.6 parts of glacial acetic acid. The commercial petroleum products require the following volumes of acetic acid for solution :

*Ber **16**, 1069.
†Quantitative Chemische Analyse durch Electrolyse 2 Aufl.

1 vol.	Common petroleum	requires	8–16	vols. glacial acetic acid.
"	crude oil (Erdöle)	"	15–30	" " " "
"	blue oil	"	25–50	" " " "
"	green oil	"	30–60	" " " "
"	petroleum residue	"	20–50	" " " "

5–20 cc. of the substance is shaken with 100–200 cc. glacial acetic acid and the separated paraffine filtered off and washed 2 or 3 times with the same solvent and then 2 or 3 times with alcohol of 75° Tralles. The remaining paraffine is dried on the filter and weighed, or dissolved in light petroleum, filtered, the light petroleum evaporated and the residue weighed.

Microscopic Examination of Powdered Roots, etc.—E. Borgmann, Ztschr. anal. Chem. **27**, 31.

The powdered sample to be examined is shaken with a little water—1 gm. root to 10 cc. water—and the mixture poured quickly upon an unglazed porcelain plate. The water sinks into the plate rapidly and leaves the fragments of the root adhering to the plate as separate distinct particles which are in a more satisfactory condition for examination than they can be obtained in any other way.

Estimation of Tartaric Acid in Argols, etc.—N. v. Lorenz, Ztschr. anal. Chem. **27**, 8. The author uses a modification of Goldberg's method as follows:

15 gms. argols or wine lees or 7.5 gms. calcium tartrate is pulverized as finely as possible and boiled in a porcelain dish of 700 cc. capacity directly over the lamp with 250 cc. water and 6 gms. dry potassium carbonate. The boiling is kept up for 20 minutes, replacing the evaporated water, and the solution is then tested for its reaction. If it is neutral more potassium carbonate must be added and the boiling continued. The solution is allowed to cool, poured into a half liter flask and made up to the mark with water. Of this solution, filtered through a dry filter, 100 cc. is evaporated in a porcelain dish to about 5–8 cc., or until there is danger of the separation of saline crusts. While still warm 5 cc. of glacial acetic acid is added and, after standing 5 minutes, 100 cc. absolute alcohol. The solution is now stirred constantly for 2 minutes and after standing for 15 minutes filtered and washed with 100 cc. of the same alcohol. The filter with its contents is

now brought into the dish in which the precipitation was made, 200 cc. water added, the dish heated until the contents boil and titrated with sodium hydroxide, which must not be stronger than 0.3 normal, litmus being the indicator. The alkali is added until the litmus changes to dark red, then the solution is boiled for 5 minutes and alkali added until the liquid has lost all trace of violet color and has a faint blue tint. The litmus solution must be carefully prepared as follows:

The best Holland litmus is ground, boiled with water and, after standing several days, the solution carefully decanted from the sediment and filtered to free it from the remaining suspended material. The filtered liquid is allowed to stand for 14 days and then carefully siphoned off from the deposit. The liquid is now evaporated somewhat and made slightly acid with hydrochloric acid, and evaporated further to free it from all CO_2. The liquid is now neutralized carefully with caustic potash solution, free from CO_2, evaporated until a strip of paper dipped in the liquid and dried appears black, and then the neutrality again very carefully adjusted. A drop of this solution colors unboiled distilled water red, and boiled distilled water violet. The potassium chloride present is without influence.

The author has investigated the process given above.

1. To see whether any free tartaric acid is formed by adding an excess of acetic acid to the potassium tartrate. A solution of neutral potassium acetate was used containing an amount of tartaric acid equivalent to 38.15 cc. of the caustic soda solution. With 50% acetic acid the following amounts were used:

cc. 50% Acetic Acid, .	3	4	5	6	10
cc. Caustic Soda . . .	37.7	37.5	37.6	37.6	37.9

With Glacial acetic acid:

cc. Glacial Acetic Acid	3	5	10
cc. Caustic Soda	37.3	37.9	38.4

The results show that no free acid is formed; but that where a very large excess of glacial acetic acid is taken the cream of tartar crystals enclose acetic acid. A very small excess of acetic acid is not sufficient, however, to decompose potassium tartrate completely.

2. He finds that the presence of potassium acetate has little or

no influence upon the result. This is contrary to the statement of Weigert[*] who directs that only a very slight excess of K_2CO_3 should be present.

3. He finds, like Kaemmer,[†] that 20 minutes boiling with an excess of K_2CO_3 is sufficient to dissolve all the tartaric acid in the substance to be decomposed.

4. That 15 minutes is sufficient time for the deposition of all bitartrate, provided the solution has been well stirred.

5. That it makes very little difference whether the alcohol used be absolute or 95 per cent.

6. That the process gives results which are too low, and that a correction of 0.0177 gms. or 0.59 per cent. bitartrate should be added to the figure found as an allowance for the amount dissolved by the 200 cc. alcohol used.

The Determination of Nitrogen by Means of Soda-Lime.

This method for the determination of nitrogen has been very carefully studied by Prof. Atwater and his associates (see Am. Chem. J. **9**, 311; **10**, 111, 113, [illegible], 262). The results have already been given, in part, in this Journal, Vol. 2, page [illegible]. In the first paper of the series Atwater and Woods reach the following conclusions: The results obtained are the same whether the proportions between the soda and lime in the soda lime are 1 : [illegible], 1 : 2 or 1 : 2.5. On the whole they prefer the mixture 1 : 2.5. The caustic soda is dissolved in water and the solution poured upon the pulverized quicklime in an iron kettle; the kettle is put over a hot fire and the contents stirred until the excess of water has been driven off and the mass fuses. It is ground while warm and separated into two portions, a finer, the particles of which pass through a sieve with [illegible] mm. meshes for mixing with the material in which the nitrogen is to be determined, and a coarser, from [illegible] to 2 mm. for charging the rest of the combustion tube. Johnson's soda lime, sodium carbonate and slacked lime, answers just as well, and slacked lime without any soda gave the same results. This is in accordance with the view that the ammonifying agent is superheated steam. For testing the purity of the soda lime oxalic acid and stearin answer better than sugar, as the latter

[illegible]

yields a small quantity of acids which affect the subsequent titration. The Knop and Arendt nitrogen bulbs are most convenient and are modified by blowing a small funnel upon the point; they are easier to fill from the burette when so made. As an indicator cochineal is to be preferred. The length of tube used depends somewhat upon the character of the substance to be burned. With ordinary work 36–40 cm. is best, though in many cases shorter tubes may safely be used. At least 40 parts fine soda-lime should be used for 1 part substance. The contents of the second paper by Atwater and Ball have already been reported (this Journal **2**, 334.)

In the third paper Atwood states that—

1. "Loss by incomplete decomposition would seem to be best prevented by fine pulverization of substance, thorough mixing with plenty of soda-lime, and heating until no considerable amount of charred residue is left.

2. "Loss by formation of cyanides which combine with the bases of the soda-lime does not seem to occur if the substance is mixed with enough soda-lime of proper water content and properly heated. Comparison of results of properly conducted combustions with those obtained by other methods implies no considerable escape of nitrogen in the free state.

3. "There is great danger of loss of nitrogen in volatile distillation products which escape ammonification. With some compounds, as alkaloids and leucine, this seems extremely difficult to avoid; but with the ordinary protein compounds of animal and vegetable tissues, and with the casein of milk, the experience of this laboratory implies that complete ammonification can be insured by providing for sufficient contact of the substance and its decomposition products with heated soda-lime, (i.e., with water vapor at high temperature.)

4. "This needed contact is best secured by (*a*) thorough mixing of substance with soda-lime; (*b*) use of soda-lime which contains a rather large proportion of lime, is not too fusible and does not shrink too much in heating; (*c*) carefully avoiding a channel; (*d*) providing a considerable anterior layer of soda-lime; (*e*) heating the latter to dull redness before bringing the heat to bear upon the substance, and keeping it hot until the combustion is done.

5. "While it is desirable to avoid the escape of matters which color the acid solution in the nitrogen bulb and render it turbid, the presence of these is not an indication of incomplete, nor their absence an indication of complete ammonification of the nitrogen."

There need be no fear of loss by dissociation of ammonia nor of oxidation on aspirating with air when the time of combustion does not exceed three quarters of an hour—

1. "Provided the tube and contents are allowed to cool slightly before aspirating with air, there need be no loss by oxidation.

2. "At a temperature sufficient to heat the tube only to dull redness there need be no considerable loss by dissociation.

3. "Ammonia may be dissociated and nitrogen lost by either very high heat, or by conducting the operation so slowly as to leave the ammonia exposed for a long time to heat. Very likely the danger of long exposure is heightened by lack of moisture from the anterior layer of soda-lime after the latter has been heated for some time.

4. "A vacant space in the tube (channel as ordinarily recommended) may involve serious loss. This loss is probably due to both incomplete ammonification of distillation products and to dissociation of ammonia."

Another precaution suggested by experience is the avoidance of dilute solutions in titrating, hence too much water should not be used to wash out the bulb.

Haynes finds also that a supposed source of error—too rapid flow of the gases through the nitrogen bulb, resulting in incomplete absorption—is of no importance provided there be a considerable excess of acid present.

The Valuation of Oil Cake.—Thomas T. P. Bruce, Warren, Chem. News, **58**, 211.

In drying an oil cake, the same precautions should be taken as in drying an oil or fat, for the residual oil in the cake is liable to oxidation and a thoroughly oxidized oil is practically insoluble. The better plan is to remove the oil first by some solvent and allow the water to subside in a warm place, in a closed vessel to prevent oxidation. Some idea of the oil under treatment may be obtained by passing a current of ozonized oxygen into it. A dif-

ference in the iodine absorption will enable one to distinguish easily oxidized oils from others, poppy, linseed and hempseed, for example, from rape and cotton. The two latter oils can be detected by the color of the coagulum which sulphur chloride produces, when the extracted oils are "blown" and heat is at the same time applied. Cotton seed gives a much lighter product than others, while the free fatty acids yield dark or black products. The electrical resistance of an oil extracted from a pure cotton cake is much higher than that of any other oil-cake, while rape, poppy and linseed follow in the order named. It must be remembered that the oxidized oils are inferior as heat producers. A. H. W.

Detection of Cochineal in Foods.—E. Lagorge, Chem. Zeit. Rep, **12,** 340. The substance is dissolved in water or dilute alcohol and if not already slightly acid it is acidified with 1 or 2 drops of acetic acid, care being taken to avoid any excess of acid. The solution is then shaken with amyl alcohol, which extracts the coloring matter. The alcohol is poured off and evaporated with sufficient water on a water bath. A few drops of a 3% uranium acetate solution are added to the water and a bluish green color or precipitate shows the presence of cochineal. The addition of an acid gives the solution an orange color. To detect it in wine the latter is shaken with a mixture of equal volumes of amyl alcohol and benzene or what is better, toluene, otherwise normal ingredients of the wine are dissolved and the action becomes indistinct. After shaking, the alcohol solution is poured off into a test tube and 2 cc. of distilled water and 1 drop of uranium acetate solution are added and the contents of the tube thoroughly shaken. A bluish green color in the water shows presence of cochineal.

If ammoniacal cochineal has been added to the wine the color of the lake passes from a violet red to a violet blue. Besides cochineal some other substances give lakes with uranium oxide. Natural wine gives yeast color, Campeche extract gives violet, and Holland wine, violet blue. The difficulty in removing the coloring matter from these wines renders the reaction with uranium acetate useless with them. S. C.

Detection of Cotton-seed Oil in Olive Oil.—E. Hirschsohn, Chem. Zeit. Rep. **12,** 341.

5 cc. of the oil to be tested is put into a test tube and 10 drops

of a solution of 1 gm. of gold chloride dissolved in 200 cc. of chloroform are added. After shaking, the test tube is allowed to stand in boiling water for 20 minutes, and if cottonseed oil is present a red color appears. S. C.

Detection of Saccharin.—S. C. Hooker, Ber. **21**, 3395. The test for saccharin described by Börnstein, which depends upon the formation of a fluoresceine compound when the saccharin is heated with resorcinol (see this Journal II, 332), is unreliable, since resorcinol when heated alone with sulphuric acid gives the same reaction.

To this Börnstein replies (Ber. **21**, 3396) that half a centigramme of the dried ethereal residue should be heated with double the weight of resorcinol and only 2 or 3 drops of sulphuric acid. Resorcinol alone will give a green fluorescence to the solution only when several decigrams are used.

Chemical Examination of Certain Gums and Resins.—Rowland Williams, Chem. News, **58**, 224. The author has tabulated the results of his interesting investigations on from 40 to 50 samples. The total potash absorption was determined by boiling a weighed quantity of the powdered resin for half an hour with semi-normal alcoholic potash in excess, adding a few drops of phenolphthaleine solution, and titrating with semi-normal HCl. The acidity was estimated by boiling with strong alcohol and titrating with semi-normal caustic potash, using phenolphthaleine as an indicator. Iodine absorptions were determined by Hübl's reagent. Ash was estimated by ignition in platinum crucibles and loss on drying at 212°F, by exposing weighed amounts to the heat of a boiling water-bath until the weights were constant. A. H. W.

Examination of Toilet Soap.—F. M. Horn, Ztschr. angew. Chem. 1888, 317. The dried soap is dissolved in absolute alcohol and the insoluble substance ($NaCl$, KCl, Na_2CO_3, Na_2SO_4, silicate of soda and ultramarine) is filtered off, washed with alcohol and after drying weighed as "insoluble in alcohol." This residue is then washed with water and what remains called "insoluble in water." The alcoholic filtrate and washings is mixed with double its volume of water and evaporated to about a third of its volume. More water is then added and the solution evaporated again until

all alcohol is driven off. The fatty acids are now separated from this solution in the usual way and the acids soluble in water extracted from the filtrate by shaking with ether.

This method prevents loss caused by the presence of extractive substance which accompanies the aromatic substance often added to toilet soap.

The Calorimetric Bomb as a Combustion Tube for Ultimate Analysis.—Arnold Eiloart, Chem. News, **58**, 284.

The Calorimetric bomb consists of a strong lidded crucible made of platinum cased in steel, with a stopcocked tube passing through the middle of the lid and near one side of it an insulated platinum wire, the lid to screw air-tight on to the crucible.

M. Berthelot records a number of interesting experiments with the use of this bomb by which combustions were made of many organic bodies, but makes no mention of it as a method of ultimate analysis; but the author questions whether it may not be practible to use it as such. He has not yet, however, been enabled to make a satisfactory investigation of the matter. A. H. W.

Detection of Iron in Oil.—B. Embe. Ztschr. angew. Chem. 1888, 362. The oil used in Turkey red dyeing frequently contains iron, and as this destroys the purity of the color obtained its detection is important. The oil is shaken in a stoppered cylinder with water containing sulphuric acid and a few drops of potassium ferrocyanide solution. Ether is now added and the shaking repeated. The oil dissolves in the ether and on coming to rest, at the junction of the two layers, a deposit of Prussian blue can be seen, if iron is present.

Estimation of Paraffine, Ceresine and Mineral Oils in Fat and in Wax.—F. M. Horn, Ztschr. angew. Chem. 1888, 458. 5–6 gms. of the substance are treated with 2–3 gms. caustic soda or potash dissolved in 80 cc. absolute alcohol. The saponification takes place very rapidly; the mass is evaporated to dryness. The dried mass is placed in a cartridge made of filter paper and the portion adhering to the dish removed with the spatula as completely as possible. The cartridge is placed in a Soxhlet's extraction apparatus aud the dish cleaned out with chloroform and these washings poured into the Soxhlet's extractor, which is then

closed and the mass extracted with chloroform, which dissolves out the hydrocarbons, leaving the soap undissolved. The solution is filtered if necessary, evaporated to dryness and heated for two hours at 105–110° to drive off the last trace of chloroform, and the residue weighed. In this way the author has succeeded in determining the paraffine in "composition candles." In the estimation of paraffine in wax it is to be remembered that beeswax contains over 50% of unsaponifiable constituents (myricyl alcohol) which are soluble in chloroform. In this case the mixture of paraffine and myricyl alcohol is boiled with acetic anhydride which forms an ester with the latter and dissolves it, while the paraffine remains as oily drops upon the surface of the liquid. The solution is filtered through a thick paper filter, placed in a hot water funnel, and the paraffine washed, first with acetic anhydride and then with water. If care is taken to keep the filter wet there is no danger of a loss of paraffine. The paraffine left on the filter is dissolved in light petroleum or chloroform, the solution evaporated and the residue weighed as before. Glacial acetic acid may be used instead of the anhydride, but in this case the myricyl alcohol separates from the filtrate on cooling.

If resin is present, as almost always happens when paraffine is used to adulterate wax, the paraffine is nevertheless obtained pure by the above process.

Estimation of Glycerine in Commercial Glycerine.—Benedict and Cantor, Ztschr. angew. Chem. 1888, 460. The authors use for this purpose the "acetin process" founded upon the following facts: When glycerine is boiled with acetic anhydride it is converted completely into triacetin. If we dissolve the mass in water and neutralize the free acetic acid with caustic soda, then add a measured excess of caustic soda and boil, the triacetin is decomposed and the excess of caustic soda may be determined. From the soda used in the decomposition the amount of glycerine may be computed. The solutions needed are:

1. *Normal Hydrochloric Acid*, normal to half normal. The strength must be determined with the greatest care.

2. *Dilute Caustic Soda* containing not more than 20 gms. sodium hydroxide per liter.

3. *Concentrated Caustic Soda*, about 10%. 1–1.5 gms. of the glycerine is weighed in a wide necked round bottomed flask of about 100 cc. content, 7–8 gms. acetic anhydride added and about 3 gms. anhydrous sodium acetate, the flask connected with a reflux condenser and the mixture boiled 1–1½ hours.

After cooling, 50 cc. water is added and the flask connected again with the reflux condenser and heated nearly to boiling. If the oil which separated upon the bottom of the flask has completely dissolved, the solution is filtered into a 400–600 cc. flask, to separate a white flocky precipitate consisting of organic impurities from the crude glycerine. The filter is well washed, the filtrate allowed to cool, phenolphtaleine added and the solution exactly neutralized with the dilute caustic soda. The proper point is reached when the yellow color of the liquid changes to a faint reddish-yellow. If it becomes really red, the proper point has been overstepped. 25 cc. of the strong caustic soda is now added and after boiling for 15 minutes the excess of caustic soda is determined by titration with the hydrochloric acid.

In the same way the amount of mono and diglycerides in fats may be determined.

On Some New Tests for Tannic and Gallic Acids.—S. G. Rawson, Chem. News, **59**, 52.

Young's test, viz. potassium cyanide, gives a reddish color with gallic acid but none with tannic acid. A precipitate forms but very slowly when a solution of tannic acid is treated with ammonium chloride; but the addition of ammonia causes at once a white precipitate which rapidly changes to a reddish brown. The same reagents give no precipitate with gallic acid; but the liquid becomes red. In very dilute solutions, the test is better applied by continuously dropping the mixture of ammonium hydroxide and chloride on the surface of the tannic acid solution, when the white precipitate may be clearly recognized, even when there is but 1 part of tannic acid to 20,000 parts of water; and if a piece of black paper be placed behind the test tube, 1 part in 50,000 of water may be detected. 1 part of gallic acid in 100,000 of water may be recognized, for while no precipitate forms, a ring, usually green on its lower surface is produced. First adding chlorine water and

then ammonia to a solution containing one of the acids is also a delicate test, a beautiful red color appearing. A mixture of potassium ferricyanide and ammonia likewise changes solutions of the acids to a dark red color. A. H. W.

PHYSIOLOGICAL CHEMISTRY.

New Reaction for Sugar in Diabetic Urine.—Marson, Chem. Centralblatt **58**, 1362. To 8 cc. urine 0.10 gms. ferrous sulphate is added, the solution heated, 0.25 gms. caustic potash added, and the heating continued for several minutes. If the urine is rich in sugar (Sp. gr. 1.030-1.060) the precipitate has a dark-green color changing slowly to black. After this deposits the supernatant liquid is brownish-red to black, according to the amount of sugar present. Urine which contains 3-6 gms. sugar per liter gives dark-green precipitates and a more or less strongly colored liquid. With normal urine the precipitate is greenish-brown and the fluid colorless. If albumen is present this is first removed by boiling and filtering.

Detection of Iodine in Urine.—C. Schwarz, Pharm. Ztg. **32**, 631. 10 cc. of the urine in question are mixed with 2 cc. of dilute sulphuric acid, (1 : 5) and to this are added 5 cc. of a 1% freshly prepared starch solution. The contents of the vessel are well stirred and a 1% solution of potassium nitrite is run in drop by drop from a burette, the stirring being repeated after each drop. If iodine is present a color ranging from a violet to a blue appears. This test is extremely delicate but a still more sensitive one is secured by using carbon disulphide instead of the starch solution. In using this a few drops of carbon disulphide must be added to the solution after each drop of potassium nitrite and the mixture well stirred as before. The author was unable to detect iodoform in urine by Lustgarten's potassium-phenol reaction but obtained the iodine reaction by using the potassium nitrite solution and obtained it also from urine where iodoform collodion had been penciled on the skin. S. C.

The Absorption of Lead Chromate.*—John Marshall, Therapeutic Gazette, **12**, 93. Lead chromate was fed to dogs and the

*See this Journal, Vol. 1, p. 358.

urine, liver, kidneys, gall and heart examined for lead and chromium. In the first experiment both metals were found in the urine after 10 days had elapsed. Lead was found in the liver, kidneys and heart; not found in the gall. Chromium was found in the liver and kidneys; not found in the heart and gall. The total amount of lead chromate administered was 7.02 gms. during 22 days. In a second experiment lead and chromium appeared in the urine after 21 days, and after 46 days lead and chromium were found in the liver, kidneys and heart; not found in the gall; total amount $PbCrO_4$ administered 77.14 gms. In a third experiment both metals appeared in the urine after 13 days, and after 23 days lead and chromium were found in the liver and kidneys; not found in the heart and gall. The author thinks that the blue line in the gums so often noticed in cases of lead poisoning arises from the action of sulphuretted hydrogen, from decaying food remaining between the teeth, upon the lead compound in the tissues. The lead chromate is decomposed in the stomach into lead chloride and chromic chloride, chlorine being also set free, $2PbCrO_4 + 16HCl = 2PbCl_2 + Cr_2Cl_6 + 8H_2O + Cl_6$ and the lead is absorbed in the stomach. The chromium is absorbed in the stomach as chromic chloride and in the intestines as sodium chromate.

The author finds that lead carbonate and phosphate are dissolved by potassium iodide solutions even in presence of sodium carbonate; and he is of the opinion that in cases of chronic lead poisoning lead is deposited in the bones and tissues as carbonate and phosphate, and not as albuminate.

The methods used in testing for lead and chromium are described by the author as follows:

"To determine the presence of lead in the urine, the method of decomposing the organic matter in the urine by means of hydrochloric acid and potassium chlorate, and separating the lead by electrolysis, as described by Lehman* was employed,—namely, concentrated hydrochloric acid was added to the urine in the proportion of about 10 cc. to 100 cc. urine. The mixture was heated, and kept at the point of gentle ebullition, and from time to time small portions (about 0.2 gms.) of potassum chlorate were

*Zeit. f. physiolog. Chem. Bd VI, p. 7.

added, until the liquid possessed a pale straw color and the odor of free chlorine was strongly perceptible. "The heating was continued, without further addition of potassium chlorate, until the odor of free chlorine had disappeared. On cooling, a partly flocculent material separated, and was filtered off, and the filtrate diluted with water to a volume equal to the original volume of the urine employed. This filtrate was placed in a glass tube of about 3-5 cm. diameter and 20 cm. length, the end of the tube being covered with parchment-paper, which was tightly bound by twine to the tube. A new piece of parchment-paper was used in every experiment. Both the twine and parchment paper were free from lead and it may be well to state here that all the reagents and filter paper used in the work were proved beforehand to be free from lead and chromium. A glass tripod, on which lay a piece of platinum foil about 2.5 cm. square, serving as a positive pole, attached to a platinum wire, was placed in a glass vessel containing distilled water very slightly acidulated with chemically pure sulphuric acid, the liquid covering the platinum foil. The parchment-covered tube containing the urine to be examined was set on the platinum foil, on the tripod, the parchment-covered end being placed directly over the foil. Another piece of platinum foil, purposely cut round so as to nearly equal the calibre of the parchment covered tube, attached at the centre to a wire (which wire for the purpose of insulation, passed through a glass tube about 25 cm. in length, and sealed at both ends) was put into urine, so that the platinum foil in the tube, serving as a negative pole, should lie on the parchment paper, as nearly as possible over the positive platinum pole, which lay under parchment paper on a glass tripod. The wires were connected with a battery consisting of three Grove cells. The current was kept up from 2 to 6 hours, dependent upon the quantity of lead in the solution. Sometimes, because of the considerable amount of lead in the solution, it was necessary to run the current only one hour. A dark gray or black deposit on the electrode in the urine would indicate the probable presence of lead.

"The electrode upon which the deposition occurred was taken out of the urine without interrupting the electrical current, and washed by gently dipping it several times in distilled water. The

foil was then placed in a small beaker, covered with nitric acid, and, after warming 5 or 10 minutes on a water bath, it was taken out, washed with distilled water, and the wash water collected in the beaker containing the nitric acid. The nitric acid solution was evaporated to dryness, the residue dissolved with a few drops of dilute potassium hydroxide solution, * and then slightly acidulated with acetic acid. The solution thus prepared was tested by special tests for lead. * * * * * * * * * * "In examining the liver, kidneys and heart for lead, they were cut in small pieces, placed in a porcelain dish, covered with water, hydrochloric acid added in somewhat larger proportion than in the case of the urine, heated to and kept at the simmering point, and potassium chlorate added in small portions at a time until the tissues were completely disintegrated. "Heating was continued, at the same time keeping up the volume of the liquid by the addition of water, until all free chlorine was driven off. The liquid was filtered and the filtrate divided into two portions. One portion was employed for the electrolytic separation of the lead, in the same manner as described in examining the urine for that metal; the other portion was examined for chromium by precipitating the lead with sulphuretted hydrogen, filtering, boiling filtrate to expel sulphuretted hydrogen, and to concentrate the volume of liquid, and precipitating the chromium by ammonium hydroxide. The precipitate was collected on a filter, washed, dried and tested with a borax bead. The obtaining of a green colored bead, the color of which was intensified by fusing a small piece of tin foil with it, was considered indicative of the presence of chromium.

"The urine, after breaking up the organic matter with hydrochloric acid and potassium chlorate, was tested for chromium in the manner described in the examination of the liver and other solid organs for the same metal. The gall was treated in like manner, both in the examination for lead and for chromium. On account of the consistency of the gall, it was diluted with water before decomposing with hydrochloric acid and potassium chlorate."

Detection of Free Hydrochloric Acid in Gastric Juice.—J. Boas, Chem. Zeit. Rep. **12**, 163.

*The potassium hydroxide employed was free from lead and iron.

Some filter paper is soaked in a saturated alcoholic solution of tropaeoline and dipped into the solution to be tested. If free hydrochloric acid is present the paper will be colored brown. If the filter paper be placed in a dish and the solution to be tested poured over it and then heated, the solution will gradually change from a dark brown to a cherry red, while around the rim of the dish the characteristic lilac color will appear. S. C.

Detection of Sugar in Urine.—H. Schnurpfeil, Chem. Zeit. Rep. **12**, 238.

The author claims that the use of lead acetate is unsatisfactory in the treatment of urine for very small amounts of sugar. S. C.

A New Reaction for Uric Acid.—By Denigès, Chem. Zeit. Rep. **12**, 238.

To a very small quantity of uric acid in a porcelain evaporating dish is added two drops of water and one or two drops of nitric acid or bromine water. This is slowly evaporated so that the residue has a yellow and not a brick red color. The residue is then dissolved in two or three cubic centimeters of concentrated sulphuric acid and mixed with a few drops of commercial benzene which gives a blue color but changes to a brown as soon as the benzene evaporates. On further addition of benzene the blue color reappears. This coloration is due to the action of thiophene on alloxan and its derivatives obtained by the oxidation of uric acid. Chemically pure benzene gives no coloration. S. C.

Ptomaines and Their Genesis in Relation to the Sepsin of Panum.—John M. Wyborn, Chem. News **59**, 2. In 1865, Prof. Panum, of Copenhagen, proved that albuminous substances, by putrefaction, yield a poisonous body, soluble in water, insoluble in alcohol, and capable of withstanding a temperature of 100°. Bergmann, who further investigated the matter, described the compound, *sepsin*, as generated by putrefaction. The author obtained a supply by the following method: Finely minced lean beef was macerated in twice its weight of drinking water, without free access of air but exposed to heat and light of the sun for sixteen days (from June 30, 1888) of dull weather with occasional sunshine. After straining, the liquid was then precipitated by an equal bulk of absolute alcohol, the precipitate was collected, boiled for five minutes in same amount of alcohol as at first used, recol-

lected, washed with boiling alcohol and dried in a warm air chamber. It was next extracted with distilled water, filtered, and evaporated to dryness on a water-bath. The residue (only 0.067 per cent of the beef used) consisted of three parts, (1) a brownish red extract, rather heavy, freely soluble in water, (2) a pale, greenish extract, very light, only slightly soluble in water, (3) brilliant white crystalline plates. Each grain (0.0648 gm.) of this residue was treated with 100 minims (5.9 cc.) of a mixture of one volume of distilled water to two of glycerine, which formed after standing, an amber colored solution with a brown precipitate. At the expiration of a month, 5½ to 6 minims or less than $\frac{1}{16}$ of a grain, administered subcutaneously, was found to be a fatal dose for mice in about an hour. The filtered solution, at first nearly neutral, after exposure to the air becomes strongly acid and has the odor of stale pepsin. Platinic and auric chlorides, tannic, picric, phosphotungstic and phosphomolybdic acids all produce precipitates in its solution. With potassium ferrocyanide, on the addition of ferric chloride, a blue precipitate appears after some time. With mercuric chloride and lead acetate, it gives white precipitates, with potassium cadmium iodide, a yellow, and with potassium bismuth iodide, a red precipitate. Boiled with potassium hydroxide, an impure ammoniacal odor is observable. Moist silver oxide is reduced to the metallic state when digested with it. With Nessler's reagent, it gives a yellowish precipitate insoluble in dilute hydrochloric acid, and with Millon's reagent, a white precipitate. Silver nitrate causes a white precipitate, soluble in ammonia, insoluble in nitric acid. A precipitate, partially soluble in nitric and hydrochloric acids, is obtained with acetic acid and potassium ferrocyanide. The dry residue, treated with sulphuric acid and potassium bichromate yields only a faint rose tint, soon vanishing. Nitric acid colors it yellow after standing several hours. From the reactions given, the author concludes, that while this body is identical with the peptones in many of its characteristics, it also shows presence of one or more of the cadaveric alkaloids either themselves insoluble in alcohol, or in combination with some substance which renders them so, and we may reasonably infer their pre-existence, unless alcohol is proved to be sufficient to produce them in the putrid solution. A. H. W.

Test for Sugar in Urine.—H. Hager, Chem. Zeit. Rep. **13**, 9. The author claims this to be the only reliable preliminary test for sugar in urine. The solution is made up as follows: 10 parts of subnitrate of bismuth, 10 parts of tartaric acid, and 50 parts of water are mixed with enough caustic potash solution to make a clear liquid and then diluted with an equal bulk of water. The urine to be tested is slightly acidified with acetic acid to precipitate the albumen which is filtered off, and to 6 cc. of the filtrate is added 1 cc. of the above reagent and the liquid is heated. If sugar is present the bismuth will be reduced and give a black color to the solution. S. C.

The Reaction of Phenol and Similar Compounds with Chloroform and Alkali.—A Raupenstrauch, Chem. Zeit. Rep. **13**, 9. The author has gone over the entire number of the different phenols and similar compounds with this reaction of Guareschi* by heating 2 cc. of a solution of the phenol in chloroform (1:1000) with a piece of caustic potash and then diluting as long as the reaction can be observed. Carbolic acid gave a light red color when diluted. 1:60,000 Ortho cresol, lilac red, until 1:80000. Meta cresol more of an orange red. Thymol, beautiful purple red with a tinge of violet until 1:20000. Guiacol, cherry red with a tinge of blue changing into a violet blue until 1:100000. Resorcinol, cherry red until 1:500000. Beta naphtol blue, changing to green and brown until 1:80000. Salol, the same as carbolic acid. Betol, the same as naphtol. S. C.

Detection of Indican in Urine.—L. Renault Chem. Cent. **59**, 500. The urine is mixed with equal volumes of hydrochloric acid and chloroform and a saturated solution of chloride of lime added drop by drop. With patients to whom iodine has been given, sodium thiosulphate is added after the chloride of lime solution. This absorbs the iodine and allows the pure blue color of the indigo to be seen.

Detection of Albumin in Urine.—Georg John, Chem. Cent. **59**, 501. The author uses a special apparatus for the examination by means of Furbringer's reaction. This consists of a small glass cylinder which is nearly filled with the filtered urine. The stopper has two clamps let into it, in which are fastened the two strips of

reagent paper—one saturated with concentrated citric acid solution, the other with potassium ferrocyanide. The chemicals in the papers are dissolved and mixed by shaking the cylinder. The paper strips are made of parchment paper, in order that fibers may not be detached.

Detection of Acidity in Gastric Juice.—F. Haas, Chem. Cent. **59,** 501. For the detection of free hydrochloric acid in gastric juice, the phloroglucin-vanilline test is to be recommended as the sharpest. This is not seriously affected by the presence of albumen, peptones, acid sodium phosphate or common salt. Congo red paper is also valuable for rapid detection of free acids.

In the examination of gastric juice the following method should be used: The filtered gastric juice is tested with Congo paper for the presence of free acid; if this gives a positive result the tropeoline reaction* is tried; if this also gives positive results we may safely conclude that more than 1 % of free hydrochloric acid is present. If tropaeoline paper gives no reaction, the bilberry color and phloroglucin-vanilline tests may be tried, with the aid of which an amount of acid corresponding to 0.25–1.% may be detected. If these give a negative result, it is safe to conclude that free hydrochloric acid is not present.

Estimation of Albumen.—Ch. Lecerf, Chem. Cent. **59,** 503. For the estimation of albumen in urine, which should not be evaporated to dryness, the author determines the nitrogen by Kjeldahl's method and calculates from this the amount of albumen present. 50 cc. urine are precipitated with sodium sulphate and acetic acid, the washed precipitate, together with 1 cc. mercury and 25 cc. pure concentrated sulphuric acid, brought into a 250 cc. flask. The flask is heated for 45 minutes, diluted with water and the ammonia, after the addition of caustic soda, distilled off in Schlösing's apparatus. The ammonia is collected in titrated sulphuric acid, etc., and the albumen calculated by multiplying the nitrogen found by 6.24.

NOTES.

L. M. Norton and H. A. Richardson find that linoleic aud ricinoleic acid volatilize readily and that they may be distilled under

*See p. 114.

reduced pressure. Neither of these so called acids appear to be chemical individuals—they are probably mixtures of two or more acids. (Am. Chem. J. **10,** 57.)

—Hempel finds that anhydrous magnesium chloride may be obtained by evaporating the aqueous solution, in a current of hydrochloric acid gas. According to the same author crystals of sal ammoniac and ammonium carbonate may be formed into solid cakes by compressing the crystals in an hydraulic press (Ber. **21,** 897.)

—Dr. G. Krüss finds that the supposed elements nickel and cobalt are really alloyed with a third element. This may be separated by treating the metals with gold chloride and washing the separated gold perfectly; it is then dissolved and precipitated again from a neutral solution with sulphurous acid. The filtrate from the precipitated gold contains the new element. The hydroxide is almost white and is insoluble in ammonia. The sulphide is black (Chem. Zeit. **13,** 113.)

—The Chemical Laboratory of the Ohio State University at Columbus was totally destroyed by fire early on the morning of the 12th instant [Feb.]. The loss is estimated at $50,000. The firemen had great trouble in saving the main building. The origin of the fire is unknown. The laboratory contained the outfit for the Department of Mining Engineering and Metallurgy. *E. and M. Jour.*

—Carbutt prepares photographic plates in which thin films of celluloid are substituted for glass. The plates are much lighter (about $\frac{1}{15}$ as heavy) and can be treated exactly like glass plates. These plates are much less liable to break than those made of glass and are so thin that they can be printed from the reverse side if the precaution is taken to use only vertical rays. (see Jour. Frank. Inst. Dec. 1888.)

OBITUARY.

—Prof. Richard Vine Tuson, for twenty-eight years Professor of Chemistry in the Royal Veterinary College, London, died at his home at Erith, Oct. 31, 1888, at the age of fifty-six. He was a

*Ber. 1872, **5,** 1055.

skilled chemist as well as a thorough teacher, and contributed considerably to scientific literature. A. H. W.

—On Nov. 16, 1888, Henry W. T. Gutzeit, Professor of Chemistry in the University at Jena. Prof. Gutzeit is known by his work on plant analysis and in pharmaceutical chemistry. S. C.

NEW BOOKS.

SELECT METHODS IN QUANTITATIVE ANALYSIS—By Byron W. Cheever, Parts 1 and 2, 2d Edition, 12mo., 700 pages. Ann Arbor, Mich., Register Printing and Publishing House, Price $2.00. This book was evidently written by Prof. Cheever for his classes, and as a time saver will be appreciated by teachers of chemistry. It comprises a list of the elements with atomic weights; rules for weighing; specific gravity determinations; preliminary steps to quantitative analysis, (sampling, grinding, drying, etc.); gravimetric determinations ($BaCl_2$, $MgSO_4$, $FeSO_4$, $CaCO_3$, Na_2HPO_4, As_2O_3); volumetric analysis; gravimetric separations; water analysis. Part II, contains directions for the analysis of limestone, iron ore, coal, blast furnace slag, iron and steel, titanic iron, chrome iron ore, lead slags, copper ore and its products, zinc ores, arsenic ores and products, manganese ores, insoluble silicates and superphosphates and the preparation of reagents.

CHEMICAL LECTURE NOTES.—By Peter T. Austen, Ph. D., F. C. S., 12 mo., 98 pp., John Wiley & Sons. This is evidently intended as a text book upon what is generally called Theoretical Chemistry. Into the middle of the book the author has injected a chapter upon "The Chemical Factor in Human Progress." This consists of an argument to show the value of chemistry to the human race and occupies 27 pages. Here is an extract from the chapter on affinity:

$$H_2 + Cl_2 = 2HCl.$$

Why should an atom of Cl leave its fellow and take up with the atom of H, indeed, take it away from its partner? The fact may be made more vivid by taking a familiar example. Let us assume that HH represent a pair of young ladies from the High School and ClCl a pair of College Students, and that these two pairs are approaching each other on a public thoroughfare. The symbols will then give a fairly good glyptic representation of the disintegration and the re-integration, union or combination of the constituents of the pairs.

* * * * * *

$$HBr + Cl = HCl + Br.$$

Assume, as before, that H represents a young lady from the High School, Br a bore, and Cl a College Student and the mechanism of the reaction becomes at once apparent."

MICROSCOPICAL PHYSIOGRAPHY OF THE ROCK-MAKING MINERALS.—H. Rosenbusch. Translation by J. P. Iddings, N. Y., J. Wiley & Sons. 8° $5.

Mr. Iddings' translation of Rosenbuch's admirable and comprehensive work, "Mikroskopische Physiographie der petrographisch wichtigen Mineralien" supplies a real want and the translator deserves the thanks of the many students with whom technical German is a source of annoyance. There are, however, several omissions from the original work, notably the absence of the colored plate. However, this plate is of little use to the beginner. The translation is an admirable abridgement, nothing being omitted that is necessary for the student's practical work and a great deal is omitted that might naturally hamper his work and could be of no possible use to him until original work were begun. The text is furnished throughout with well executed cuts and (as in the original) there are twenty-six full page plates of sections. The work of Mr. Iddings can hardly be called into use by the advanced student, nor does the author claim it to be for such use.

The translation on the whole is very praiseworthy and should, and undoubtedly will, be in the hands of every petrographer.

J. E.

CALIFORNIA STATE MINING BUREAU, WM. IRELAN, JR., STATE MINERALOGIST, EIGHTH ANNUAL REPORT.—Sacramento, J. D. Young, State Printer. This is a very useful book. In addition to the progress reports it contains treatises on the Milling of gold ores in California, by J. H. Hammond; Drift Mining in California, by R. L. Dunn; Lithology of Wall Rocks, by M. Atwood; Water Wheels, by F. F. Thomas; Notes on Western Lead Smelting by W. S. Keyes; The Russell process; Notes on the Hydrometallurgy of gold, by C. H. Aaron; Notes on the Hydrometallurgy of Silver, by C. H. Aaron; Natural and Artificial Cement; Building Stones, by C. W. Jackson; U. S. patents in California; Water Rights and the Alien Act. The book is well and fully illustrated and is a credit to the Mineralogist and to the State.

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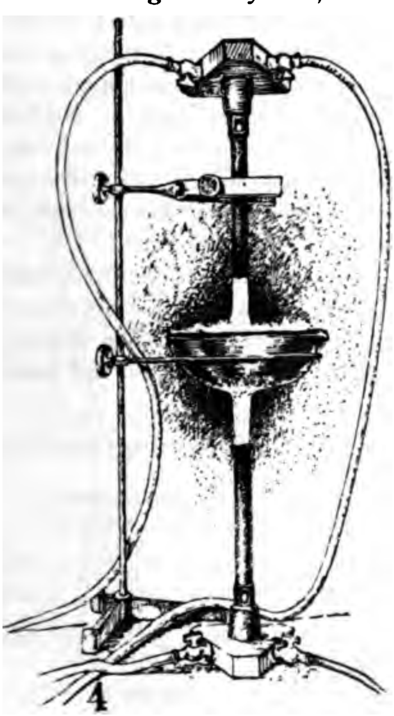
SILICON IN PIG IRON.

DETERMINATION BY A METHOD OF RAPID EVAPORATION.

By CLEMENS JONES.

In the "direct process" of making steel, and very frequently when making foundry iron, it is necessary to determine silicon in the iron in a few minutes. To do this within the time-limit with reasonable accuracy has been found a task by no means simple.



By Ford's method, silicon can be determined in twelve minutes after taking the sample. There are several modifications of this method, but each has the objection that during evaporation a loss is caused by spattering.

The endeavor to overcome this defect led to experiments on shortening the time by merely hastening the modus operandi of a standard method. Dr. Drown's admirable method was selected, and by the use of the device figured in the cut, it was found that evaporation of 50 cc. of nitro-sulphuric acid could be done in three minutes, with complete dehydration of the silica, and very little

loss, as evaporation is not carried to dryness. Moreover, the disappearance of nitric acid is marked by a strongly tinged flame reaction.

A platinum dish of 150 cc. capacity is used. 1 gm. of borings is transferred to this, 50 cc. nitro-sulphuric acid added, and the dish is then placed between the flames from two blast lamps. The arrangement of the lamps calls for no description; a slight adjustment of the flames, in both distance apart and intensity, is all that is required before the right effects are secured. This being done, however, the arrangement becomes a fixture. The downward flame should be a trifle stronger than the upward one. In fact, wire gauze beneath the dish gives better results, by distributing the heat evenly. A Bunsen burner may be substituted for the lower lamp. Violent ebullition is prevented by the current of heat and air upon the surface of the boiling liquid, so that by giving the dish a circular motion with a pair of tongs, when nearing final expulsion of nitric acid, the fumes of sulphuric acid are quickly obtained, accompanied by a sharp play of reddish yellow color of the impinging flame. When finished, the sulphate of iron is in a thick, semi-pasty state, into which hot water can be poured at once. Again placed between the flames, this is speedily dissolved and is ready for subsequent treatment.

I may add that gasolene gas was employed in the experiments.

A few comparative results are given below. The irons chosen were 1 low, 2 medium, and 1 high silicon iron. The slightly higher results by rapid evaporation are due to undissolved ferric oxide formed by the intense heat.

No. 2602. Reg. Method (Dr. Drown's). Silicon = 0.545 per cent.

Rapid Evaporation,

	cc. Acid.	*Time for Evaporation.*	*Per cent. Silicon.*
1.	50.	5 minutes.	0.657
2.	50.	4 "	0.639
3.	50.	4 "	0.629
4.	25.	2½ "	0.639

No. 3252. Regular Method. Silicon, 3.858 per cent.

Rapid Evaporation.

	cc. Acid.	*Time for Evaporation.*	*Per cent. Silicon.*
1.	50.	5 minutes.	3.863
2.	50.	5 "	3.849
3.	50.	3 "	3.765

No. 2074.	Regular Method.	Per cent. Silicon.
1.	"	2.374
2.	"	2.384

Rapid Evaporation.

	cc. Acid.	Time for Evaporation.	Per cent. Silicon.
1.	50.	5 minutes.	2.374
2.	50.	5 "	2.328
3.	50.	5 "	2.276
4.	50.	4 "	2.320

No. 1967. Regular Method. Silicon 1.829 per cent.
Rapid Evaporation.

	cc. Acid.	Time for Evaporation.	Per cent. Silicon.
1.	50.	5 minutes.	1.955
2.	50.	4 "	2.058
3.	50.	4 "	2.010
4.	50.	5 "	1.810
5.	25.	3 "	1.904
6.	50.	3 "	1.913
7.	50.	3 "	1.815

No. 4 of this series lost silica in igniting. No. 7 was completed in 20 minutes from the time of opening the balance case. 10 minutes was consumed in filtering, and the carbon burned off in the old way. By igniting with oxygen, and filtering more rapidly, the time for a complete determination can be undoubtedly reduced to 15 minutes.

A RAPID METHOD FOR THE REDUCTION OF FERRIC SULPHATE IN VOLUMETRIC ANALYSIS.*

BY CLEMENS JONES, HOKENDAUQUA, PA.

The difficulties attending the reduction of ferric sulphate in the determination of metallic iron by the method of Marguerite, are often serious, and affect time, patience and accuracy. They depend upon the condition and purity of the zinc used, the state of saturation of the solution, acidity, and temperature.

D. J. Carnegie (*Chem. News*, lvii. No. 1481), says that ferric solutions are instantaneously reduced by the use of zinc-dust.

* Read before the American Inst., Mining Engineers, New York Meeting, Feb. 1889.

But there are obvious objections to its employment in quantitative work. Dr. Drown found that "a concentrated solution, not too acid, was reduced by 5 grammes of pulverized zinc in two or three minutes, without other heating than that due to the chemical action." But the zinc in excess of the stoichiometrical amount must be dissolved. This has been the main obstacle. Large excess of sulphuric acid, heat, or both, must then be employed; and either involves loss of both time and accuracy in the volumetric method.

Marguerite's method is, in point of celerity and refinement, the most scientific of all the "iron methods." If the iron solutions have been properly prepared, the end-reaction is sharper and more quickly obtained than by any other. There is no loss in testing for final oxidation, as potassic permanganate is its own indicator. Besides, the permanganate solution can be most quickly prepared, and accurately standardized.

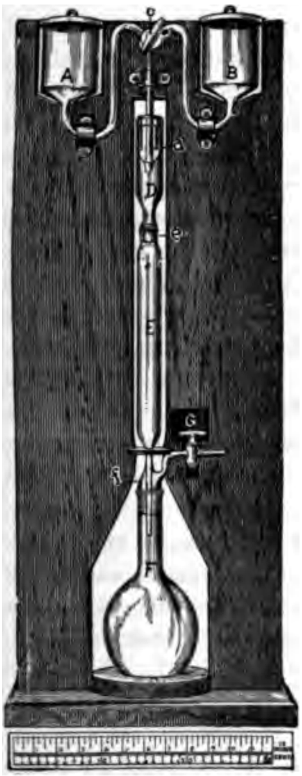
Hence there is a natural preference for this method in determining metallic iron. It permits direct and rapid titration. Only facilitate the reduction of the ferric sulphate; and it is the most valuable of all methods, combining the utmost rapidity with the highest accuracy attainable.

There is no difficulty in obtaining iron as ferric sulphate; but the reduction of this to the ferrous state has hindered the precision of the permanganate method. Discrepancies in results are all to be set down to the errors introduced in the reduction with zinc. The care to secure complete reduction—how frequently is it lost through the presence of a minute grain of zinc, washed down from the neck of a flask in titrating, and now derisively tossing up a little chain of hydrogen bubbles! Again, a very hot solution will decompose an excess of reagent. And moreover, as I shall again remark, it is probable that the largest impurity in pulverized zinc, is not iron, as heretofore supposed, but carbon.

The size of the particles of the pulverized zinc employed in the method which this paper describes is determined by passing through a 40-mesh, upon a 60-mesh sieve.

Observing the action on zinc of ferric solution in sulphuric acid, I noticed that in all cases, with practically the same amount of acid and zinc, the latter dissolved more rapidly when the solu-

tion was more highly saturated with ferric salt; that the time for reduction, under the conditions mentioned, was dependent on the strength of the ferric solution. This was evidence that a sufficient number of zinc particles should be exposed, in a proper ratio to the ferric salt; that each molecule takes the same time to reduce, when unaffected by its unreduced neighbor, which is the time of contact: and that, therefore, were each molecule presented to new particles of zinc successively, reduction would be instantaneous. Filtering the ferric solution through pulverized zinc, would then demonstrate this fact, which I found to be the case. *A solution of ferric sulphate is instantaneously and completely reduced by filtering through pulverized zinc.*



Reductor, for the Reduction of Iron Solutions by Filtration through Zinc.

The apparatus figured below was designed for the rapid reduction of ferric solutions. It consists of two cups* A, and B, of about 300 cc. capacity, the outlets of which connect alternately, by means of a 3-way cock C, into the joint-tube D. This tube is provided with a packing space *d*, which is filled with an asbestos roll, kept in place by a ring of cork. The tube moves up and down freely. At its lower end *e*, it is ground to fit the detachable tube E. This is for the pulverized zinc, and at its lower end has a stopper *f*, ground to fit the titrating flask F, of about 700 cc. capacity. The stop-cock G connects with the vacuum-pump. The tube E is disconnected by pushing it up, grasping the neck of the flask with one hand, removing with the other the round block which supports the flask, and then detaching the flask F. The tube E now rests on the little shelf shown

*I find this form of cup not so convenient as the bulb-shape of a separatory funnel of the same capacity.

in the cut. This is drawn out, and frees the tube at its ground-joint *e*.

A wad of fine glass wool, sufficiently thick to make a good filter, is inserted into the tube, and the pulverized zinc is filled, over this, for four fifths the length of the tube. This holds about 300 grammes, which is enough for 60 filtrations. The tube is then replaced by reversing the manner of removal, connection is made with the filter-pump, and the apparatus is ready for use. It is first necessary to wash the column of zinc with dilute sulphuric acid. This acid is made up of one part of acid of 1.84 sp. gr. to two parts of distilled water, and is used throughout in all work upon iron determinations. Of this about 30 cc. is taken, transferred to the cup A, and diluted to 300 cc. Stop-cock G is opened for suction, and then the three-way cock C is turned to discharge the cup A. A vacuum of 5 to 6 pounds or more, gauge-indication, must be used, since the large volume of disengaged hydrogen retards the filtration. The cup and the column of zinc are then washed free from acid by five rinsings. This operation is repeated four times, and is then finished for this tubeful of zinc.

Below is a series of trials, undertaken to determine the proper correction for iron in the zinc. Owing to the small amount of reagent required for the end-reaction, it was impossible to obtain a reading under 0.1 cc., which, as will be seen further on, is too much for even so large a quantity of zinc.

No. of Test.	Amount of $K_2Mn_2O_8$ cc.
1	0.4
2	0.2
3	0.13
4	0.12
1	0.4
2	0.2
3	0.15
4	0.13
1	0.4
2	0.15
3	0.13
4	0.11

1	0.4
2	0.2
3	0.13
4	0.1

These were made at different times upon new quantities of zinc, and with solutions of $K_2Mn_2O_8$, of slightly varying strength. Beyond the fourth trial in each case, it was impossible to get less than one drop of the reagent into the solution. This is nearly 0.1 cc. and in every instance gave a strong coloration.*

Evidently the largest amount of iron in the zinc is only on the outside of the particles, as the quantity dissolved in the reductor is less than five grammes for each filtration. By the ordinary method of dissolving all the zinc, the correction is rarely below 0.15 cc. for 5 grammes.

Exactly the same procedure is adopted to reduce a ferric sulphate solution, except to use both cups A, and B. After the first is emptied it is washed with cold water five times. This incidentally cools the column of zinc and retards unnecessary solution. The other cup is then connected by turning the 3-way-cock C, and washed in like manner. In each case this suffices to free the apparatus from all traces of iron or sulphuric acid. G is then closed, the 3-way-cock is turned again to relieve the pressure, and the flask is removed. Time for reduction, 2 minutes.

Solutions obtained in this way are beautifully clear and perfectly reduced. The volume and temperature are practically constant: the former being about 400 cc., and the latter about 40° centigrade. Uniformity marks the entire process, and there is a minimum quantity of zinc sulphate. Dilute solutions must be used. There should be not over 50 cc. sulphuric acid in 300 cc. of the ferric solution ready for reduction.

An experiment was tried in reducing the solutions, after oxidation, used in standardizing the permanganate. Iron wire taken at .996 Fe.

Standardization.

	Weight of Wire, gm.	$K_2Mn_2O_8$ cc.	Reaction.	Factor.
No. 1	0.1271	16.48	Tinged	1 cc. =.007681 Fe
No. 2	0.12175	15.78	Sharp	1 cc. =.007684 Fe

*I have since obtained a reading down to 0.03 cc. by closing the burette-tip.

Reduction of these Solutions.

	Reduction.	$K_2Mn_2O_8$ cc.	Reaction.
No. 1	complete	16.42	Tinged
No. 2	"	15.8	Sharp

This treatment applied to a hematite ore, gave (No. 2969)

	Weight of Ore, gm.	Reduction.	$K_2Mn_2O_8$ cc.	Reaction.	Iron, Per cent.
No. 1	0.1527	complete	7.2	Tinged	34.7207
No. 2	0.1526	"	7.2	sharp	34.7441
(Reverted : filtered through the Reductor)					
No. 1		"	7.2	sharp	
No. 2		"	7.2	"	

Direct determination of metallic iron in the wire used in standardizing gave the following results: weight of wire, 0.12405 gm. dissolved in sulphuric acid and oxidized. Reduced by filtration.

Reduction.	$K_2Mn_2O_8$ cc.	Reaction.	Iron, Per cent.
Complete	16.07	sharp	99.5420

Factor, 1 cc. $K_2Mn_2O_8$=.007684 Fe.

Solution again reduced by filtration. Reduction complete. $K_2Mn_2O_8$, 16.10 cc., as compared with 16.07 in the former reduction. Difference, 0.03.

The *apparent* correction for iron in the zinc, is the increase in last reading or 0.03 cc. of the reagent. Time required to reduce and titrate this solution twice, *ten minutes*.

While the apparent correction is thus 0.03 cc., it is inadvisable to use it as a constant, since the true correction can be applied for each case, and is really variable, depending upon acidity and degree of saturation.

In the following example, the test of hematite-ore (No. 2922), carried through in duplicate, the ferric sulphate was first reduced by the usual method of adding the zinc to the solution, and then entirely dissolving it, and deducting 0.3 cc. as the correction after titration. Each solution was then filtered through the Reductor, and on the supposition that for each treatment with zinc (on account of filtration), but 0.1 cc. is to be deducted; the results are seen to be coincident. By "Reverted" is meant filtering through the Reductor. The value of 1 cc. $K_2Mn_2O_8$ is .007678 Fe.

	Weight of Ore gm.	Reduction.	$K_2Mn_2O_8$	Correction.	Iron, Per cent.
No. 1	0.1360	complete	7.4 sharp	0.3=7.1	40.06
"	Reverted	"	7.3 "	0.2=7.1	= 40.06
No. 2	0.1379	"	7.49 "	0.3=7.19	= 40.03
"	Reverted	"	7.39 "	0.2=7.19	= 40.03

Another (No. 2971) with a difficulty-fusible residue, was tried directly, each solution being reduced twice by filtration. Time for reducing and titrating in duplicate, twenty minutes.

	Weight of Ore, gm.	Reduction.	$K_2Mn_2O_8$ cc.	Reaction.	Iron, Per Cent.
No. 1	0.1505	complete	9.27	sharp	
2d trial		"	9.21	"	= 47.0230
No. 2	0.1823	"	11.29	"	
2d trial		"	11.23	"	= 46.9132

Assuming that for ordinary purposes the correction obtained by reverting the solution used to standardize the permanganate would be a safe one, I have applied it to the determination of iron in an ore with the following results:

Standardization.

	Weight of Wire, gm.	$K_2Mn_2O_8$ cc.	Reduction.	Correction cc.
No. 1	0.11915	15.48		
"	Reverted	15.55	complete	0.07
No. 2	0.1261	16.4		
"	Reverted	16.44	"	0.04

Factor, 1 cc. $K_2Mn_2O_8$=.007664 Fe.

Determination.

(No. 2997)	Weight of Ore, gm.	$K_2Mn_2O_8$ cc.	Correction.		Iron, Per cent.
No. 1	0.1576	9.58	0.07	9.51	46.246
No. 2	0.2351	14.3	0.07	14.23	46.388

In like manner a rich hematite ore (No. 3066) gave:

No. 1 weight of ore,	0.2302 gm.;	Metallic iron	60.03	per cent.
No. 2 " "	0.1645 "	" "	60.17	"

Theoretically a neutral, or nearly neutral solution of ferric sulphate should be reduced by zinc in this way without dissolving the latter appreciably, at least, not to the extent of introducing the error due to its impurity. As nearly satisfying these conditions, by the use of a minimum of sulphuric acid, the two following examples afford a remarkable demonstration. The experi-

ments were conducted like the foregoing. Factor 1 cc. $K_2Mn_2O_8$ =.007656 Fe.

No. 1. Weight of Wire, 0.12085 gm.	Amount of $K_2Mn_2O_8$ cc.
1st trial	15.72
2d "	15.78

No. 2. Weight of Wire, 0.11315 gm.	$K_2Mn_2O_8$ cc.
1st trial	14.695
2d "	14.695

Metallic Iron 99.432 per cent.

There appears to be a deeply-rooted belief that iron is the only impurity in pulverized zinc which reduces the $K_2Mn_2O_8$; but I think the error is in nearly all cases largely due to the carbonaceous residue. Five grammes of zinc were dissolved in sulphuric acid, and then found to require 0.13 cc. of $K_2Mn_2O_8$. After oxidation, a test of the solution gave *no* coloration with potassium sulpho-cyanate. Oxidation with nitric acid gave a like result.

Twenty-five cc. of sulphuric acid were diluted to 300 cc., and passed through the reductor. $K_2Mn_2O_8$ required 0.05 cc. One drop more was added, and the solution allowed to stand until clear. It was then tested with potassium sulpho-cyanate, and gave *no* coloration. A like experience followed in repeated trials of dissolving the zinc directly; but subsequent treatment of these solutions with ammonia and filtration gave evidence of iron. A determination of iron in the zinc used in these experiments gave the following: 5 grammes of zinc were dissolved in sulphuric acid. $K_2Mn_2O_8$ required 0.1 cc.=0.0154 per cent. iron. As in the first case, 5 grammes were dissolved and the solution filtered through the reductor. $K_2Mn_2O_8$ required 0.12 cc. Increase, 0.02 cc., equivalent to 0.0030 per cent. iron.

Mr. P. W. Shimer has since suggested to me the probability of lead existing in the black residue. This I have found to be the case.

Ignition of the residue leaves a small fused mass, which, on dissolving in hydrochloric acid, reveals iron by the sulpho-cyanate. May not this residue be soluble under accidental conditions when exposed to the action of the solution for an hour or more by the old method of reduction? I have frequently noticed that it is perceptibly smaller in bulk. Again, if the solution is warm,

may not final oxidation of the ferrous salt have an effect in dissolving it?

If it should prove to be a carbon compound, the reducing action on the $K_2Mn_2O_8$ would be increased beyond the correction for iron as ordinarily obtained. Filtration undoubtedly obviates these difficulties.

As touching this reducing action, the following experience may be cited. An analysis in duplicate by the old method, gave an unaccountable difference in results. The one solution, with the high result, was noticed to have a dark color and luster. Subsequently this proved to be due to a very finely divided sediment, apparently carbon.

(No. 2978)	Weight of Ore, gm.	Reduction.	$K_2Mn_2O_8$ cc.	Correction.	Iron, Per cent.
No. 1	0.2174	complete	11.75	0.3	40.47
No. 2	0.1795	"	11.12	0.3	46.31
No. 2	Reverted	"	9.61	0.15	40.49

Factor, 1 cc. $K_2Mn_2O_8$=.007684 Fe.

Here is another similar case:

	Weight of Ore, gm.	Reduction.	$K_2Mn_2O_8$ cc.	Correction.	Iron, Per cent.
No. 1	0.1951	complete	16.95	0.18	65.80
No. 2	0.1928	"	15.6	0.18	60.92
No. 1	Reverted	"	15.9	0.3	61.21

Factor, 1 cc. $K_2Mn_2O_8$=.007656 Fe.

The following test of an ore is added as an illustration of the capability of this method for rapid reduction. 1 cc.=.007671 Fe.

	Weight of Ore, gm.	Reduction.	$K_2Mn_2O_8$ cc.	Correction.	Iron, Per cent.
No. 1	0.1566	complete	13.35	0.05	65.1496
No. 2	0.1820	"	15.51	0.05	65.1569

Time required to reduce and titrate in duplicate, twenty minutes.

The investigation of this principle of reduction, which I believe to be new, has been governed by the time afforded in the few and brief intervals of regular technical work. I think the method capable of still further advancement and application. At the present stage of the inquiry, it presents the most rapid and accurate treatment for the estimation of iron.

The apparatus was made by Emil Greiner, 63 Maiden Lane, New York, whose intelligent and skilful workmanship has been of great service to me.

THE DETERMINATION OF CHROMIUM IN CHROME IRON ORE.

By Leonard P. Kinnicutt and George W. Patterson.

The complete decomposition of chrome iron ore, and the determination of the amount of chromium, is one of the difficult problems of quantitative analysis. Of the very many methods that have from time to time been published, one given last year by Donath,* using barium dioxide as the oxidizing and fusing agent, seemed the most expeditious and simple.

The method as given by him was as follows: One part of the very finely divided mineral was mixed with five times its weight of barium dioxide, and heated for one half-hour in a porcelain crucible over one Bunsen lamp. The semifused yellowish green mass thus obtained was treated with dilute hydrochloric acid. After a few hours, all the chromium was found in solution as the chromate of barium. The barium was precipitated with sulphuric acid, and the filtrate from the barium sulphate neutralized with sodium carbonate, and a few drops of an alkaline solution of permanganate of potassium added to oxidize any chromium that might have been reduced. The excess of permanganate was then removed by the very careful addition of ferrous sulphate, and the amount of chromium determined volumetrically.

Donath, however, did not publish any results to show the accuracy of the method, and it was on this account that we were led to repeat his work.

A few experiments were sufficient to satisfy us that the perfect decomposition of chrome iron ore from Pennsylvania could not be accomplished in the manner described by Donath. A number of fusions of the very finely powdered and sifted mineral with five or even ten times its weight of barium dioxide were made, and in every case the residue contained a greater or less amount of the black undecomposed mineral. Also, we were unable to obtain any porcelain crucibles in which more than one fusion could be safely accomplished; while if the fusion was made in platinum crucibles, we obtained, as was also noticed by Donath, a comparatively large amount of the sesquioxide of chromium.

Another serious difficulty was met with later in the process;

*Dingler's Polytechnisches Journal, vol. cclxiii. p. 245.

namely, in determining the exact point where sufficient ferrous sulphate had been added to reduce the excess of permanganate of potassium, used in oxidizing the small amount of reduced chromic acid. The change of color of the solution being so slight as to be almost useless as an indicator.

We were therefore unable, with the most careful manipulation, to obtain satisfactory results with the above process. In our study of the process we made a great many experiments with different fusing mixtures, and also tried various ways of oxidizing the small amount of sesquioxide of chromium we always found present in the solution. The results of these experiments have enabled us to formulate a very quick and simple process, which, as can be seen by the results given below, is also, as regards accuracy, very satisfactory.

About three tenths of the very finely divided mineral is mixed with twenty times its weight of a mixture containing equal parts of dry sodium carbonate and barium dioxide, and heated in a platinum crucible with the full flame of one Bunsen lamp for one half-hour. At the end of this time a quiet fusion is obtained and the decomposition is completed. The crucible is then placed in a beaker, covered with water, and hydrochloric acid added, a little at a time, till the mass is completely disintegrated. The crucible is then removed, the solution made strongly alkaline with caustic potash, and five or six cubic centimeters of a five per cent. solution of hydrogen dioxide added to oxidize the small amount of reduced chromic acid that may be present. The solution is now boiled for twenty minntes to remove any excess of hydrogen dioxide, made acid with hydrochloric acid, and the amount of chromic acid determined by the aid of a standardized solution of ferrous chloride.

Six analyses of a sample of chrome iron gave the following per cents of chromium sesquioxide.

1.	2.	3.	4.	5.	6.
49.87	49.97	49.84	49.85	49.88	49.87

The solution of ferrous chloride used was kept under oil and titrated before each analysis. One cubic centimeter equalled about 0.015 gram of chromium sesquioxide.

SALISBURY LABORATORIES,
WORCESTER POLYTECHNIC INSTITUTE.

TYROTOXICON IN MILK.

BY LEONARD P. KINNICUTT,

On Friday evening, November 9th, Dr. George E. Francis brought me a pint of milk, and gave me the following facts:

"I was called this morning to attend five persons in one family suffering from an attack of cholera morbus. On inquiring, I found that, of the two members of the family not ill, one had taken no cream or milk, while the other had taken only boiled milk with her breakfast. The five members who were ill had all taken cream or milk of the previous day, and the symptoms of poisoning had begun about two and one half hours after breakfast."

The milk was in a pint beer bottle with patent rubber stopper, and appeared and tasted perfectly fresh and good. After carrying it to the laboratory it was allowed to remain in the tightly stoppered bottle for one week before it was examined. The milk had by that time decomposed, separating into two layers. It was filtered through thick Swedish filter-paper, the filtrate neutralized with a dilute solution of sodium hydrate, placed in a separating funnel, and shaken thoroughly with ether. A thick emulsion formed, and it was only after four days, and by the use of various mechanical means, that a separation could be effected. The ether solution was allowed to evaporate at the ordinary temperature, and the residue carefully tested. Reactions were obtained which agreed perfectly with those given by Vaughan, *Journal of Analytical Chemistry*, vol. 1, pp. 25 and 281, for tyrotoxicon.

There is therefore no doubt that the poisonous action of the milk was caused by the same poison that Vaughan found in the various cases cited by him.

A visit to the dairy from which the milk was obtained was made, and it was found that the herd consisted of fifteen Jersey cows, all in the best condition, well fed and cared for. The dairy supplies about forty families with milk, and the milk of all the cows is mixed together, before subdividing it into the various portions.

And as only one family out of the forty supplied with the milk, as far as can be found out, suffered from any poisonous effect, it proves that the poison was developed after the milk had been delivered. This was also found to be the fact on questioning the servants of the family poisoned. The milk had been received in a tin can, which it was their business to keep clean, and it had been immediately subdivided into two portions. One portion was placed in an earthen dish to raise cream, and the other was used during the same morning as fresh milk, without causing the slightest trouble. The symptoms of poisoning were caused by the first portion, after standing over night. The above facts seem to show that the tyrotoxicon was developed during the twenty-four hours after the milk was received.

The only explanation of its development that I can at present give is that the cans used for obtaining the milk had not been thoroughly scoured with boiling water, and that a little old milk remaining on the inside edges of the can had undergone a peculiar fermentation, and had caused the development of a sufficient amount of tyrotoxicon, during the twenty-four hours it had remained in a cool place, to produce the poisonous action.

I have been led to describe thus fully the above case, as up to this time almost nothing is known as to the cause of the formation of tyrotoxicon in milk. I have, I think, pointed out one way in which it may be developed, namely, the use of cans which have not been kept perfectly clean; but it is only by the careful examination of a number of cases that it will be possible to decide whether the formation of the poison is due solely to such causes.

SALISBURY LABORATORIES,
WORCESTER POLYTECHNIC INSTITUTE.

CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF VANDERBILT UNIVERSITY.

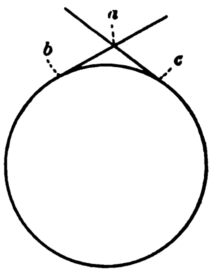
II. NOTE ON A METHOD FOR CUTTING GLASS TUBES AND CYLINDERS.

BY W. H. HOLLINSHEAD.

Several methods for cutting large glass tubes and cylinders have been employed with varying success; but the following

plan, which has been in use here about a year, has so far proved the most satisfactory.

Make a small scratch on the tube with a file at the point where it is to be broken. Then wrap around the tube a stout platinum wire, one end of which is connected with one pole of a battery of six or eight Grove's cells. It is best to have the points of tangency at *b* and *c* upon the scratch; but the ends of the wire must not touch at *a*.



When all is ready, complete the circuit. The break will follow the red-hot wire.

Should the current be too strong or the wire too small, the latter will be melted, but this may be avoided by increasing the length of wire subjected to the current. Six or eight of Grove's cells will give the requisite heat for cutting heavy combustion tubing. More cells and a stouter wire will be required for extra heavy tubes or large cylinders.

The current from a dynamo may be advantageously used, for then the current could be turned on and off at pleasure by a switch.

THE DETERMINATION OF AMMONIA.

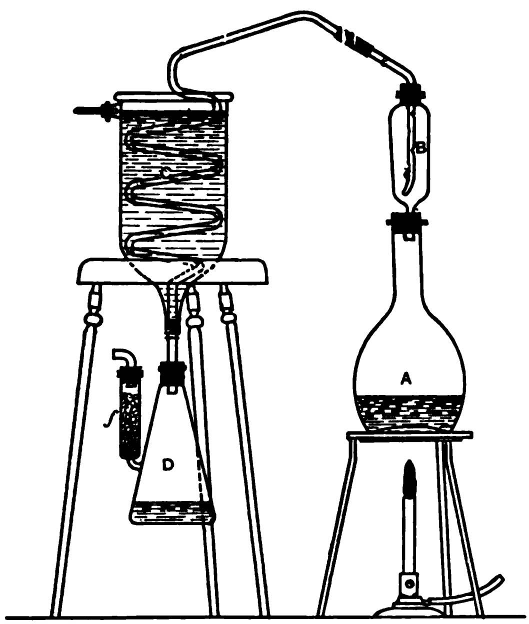
BY WALTER M. STEIN AND PAUL W. SCHWARZ.

The determination of ammonia by means of distillation with caustic potash or soda and collecting the ammonia in titrated acid, notwithstanding its apparent simplicity and theoretically almost absolute accuracy, has technical difficulties which make more complicated methods appear simpler to many chemists.

In using Schloesing's litmus preparation or other equally sensitive indicators, the titration can readily be brought to the utmost accuracy and the difficulties rest therefore exclusively with the distillation itself.

We know of no safety appliance, up to the present, which can be implicitly trusted to effect its purpose of preventing the carrying over of small particles of the alkaline solution from the distilling flask to the condenser and receiver, especially if zinc filings are used in order to produce a slight (slow) disengagement

of gases; and they are even less reliable in using the Kjeldahl method for the determination of nitrogen, when the ammonia is to be distilled off from a solution very rich in salts. For these reasons, we believe, chemists engaged in fertilizer analyses, will welcome a description of an apparatus which can easily be put together and which has been in use in our laboratory for ten months to our entire satisfaction and needing but little supervision.



The vapors escaping from the distilling flask A pass through a pear-shaped bulb B into which, through a rubber stopper, is inserted a glass tube e, to lead the vapors to the condenser C. This glass tube is slightly drawn out at the lower end and bent to one side as shown in the sketch, to prevent particles of the solution from being thrown directly into e. In that part of tube e, which reaches into B, there are three holes of nearly the same width as the tube and about one inch apart, which can be produced either by filing or by means of the blow-pipe.

As the distillation progresses, the lower end of the bulb B and also that of the tube e will soon be closed by small quantities of condensed steam, which will at the same time wash the rising vapors and prevent them from entering e from below. Owing to the openings in e, the vapors are forced to enter this tube from the side, and as there is no possibility of any pressure therein which could cause a current, any liquid which might be thrown

in through one of the holes when the boiling is very violent, will run off harmlessly through the lower end.

We find a further advantage in the receiver *D*. It is an Erlenmeyer flask, connecting at about mid-height with a perpendicular tube *f*, ¾″ in diameter, filled with small glass fragments and closed on top by a cork perforated by a narrow but open glass tube. The standard acid is to be introduced through *f*, and the small quantity held back by the glass fragments amply suffices to absorb the small traces of ammonia, which are not carried into the main part of the acid at the bottom of *D* by the condensed steam. Of course, the excess of acid is to be titrated directly in *D*, and on account of the small remnant of acid in *f* great care is not needed in adding the alkali while titrating. The flask holds, up to the tube connecting with *f*, about 250 cubic centimeters; amply sufficient for a distillation.

It is obvious that there can be no pressure at any time, nor in any part of the apparatus and consequently there will be no strain on the connections, as is the case with the receivers usually employed. The use of an aspirator, which greatly facilitates the throwing over of the liquid, and on this account must be closely watched, becomes unnecessary.

Since we have used the above described apparatus, we have been unable to trace any fixed alkali in the distillate in any single one of numerous experiments.

Mr. E. Greiner, 63 Maiden Lane, New York City, has supplied us with the essential parts of the apparatus.

TECHNO-CHEMICAL BUREAU AND LABORATORY,
323 Walnut St., Philadelphia.

REICHERT'S BUTTER TEST.

BY DR. BENNETT F. DAVENPORT.

The following minor differences are to be noted between a true butter and a straight oleo, when examined after the Reichert's method.

In the first place, if tasted, the oleo will be found to be deficient in the fullness of flavor of a true butter, while sweet; and it will never acquire the strong flavor and odor of a butter which has turned rancid.

Then when the sample is melted, to allow the curds, water and salt to settle out to the bottom, it will be noticed that the deposit of curds in an oleo will not be more than about one-quarter as much as in a butter. Again, with oleo the saponification is not quite as rapidly completed, and the soap formed is not as rapidly dissolved in the water afterwards added. And when soda is the alkali used the oleo soap forms a markedly more viscid solution with the water.

Again in the case of an oleo, a ring of solid deposit forms in the inner tube of the condenser during the distillation, which never occurs at all with a pure butter.

These differences occurring one after another, will enable the analyst to form a fairly reliable opinion as to whether the sample is oleo or butter long before the completion of the distillation.

A NEW FORM OF FAT PERCOLATOR.

By H. J. Patterson.

A modification of the ordinary percolator with its filter paper or linen bottom is shown in the accompanying figures.

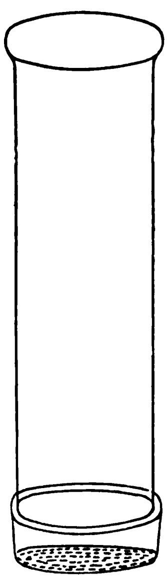


Fig. 1.

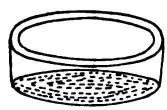


Fig. 2.

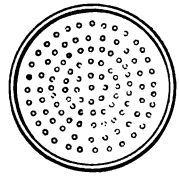


Fig. 3.

It consists of a glass tube ground at the lower portion, so as to make a perfect fit with a nickel cap which has a perforated bottom like the Gooch crucible.

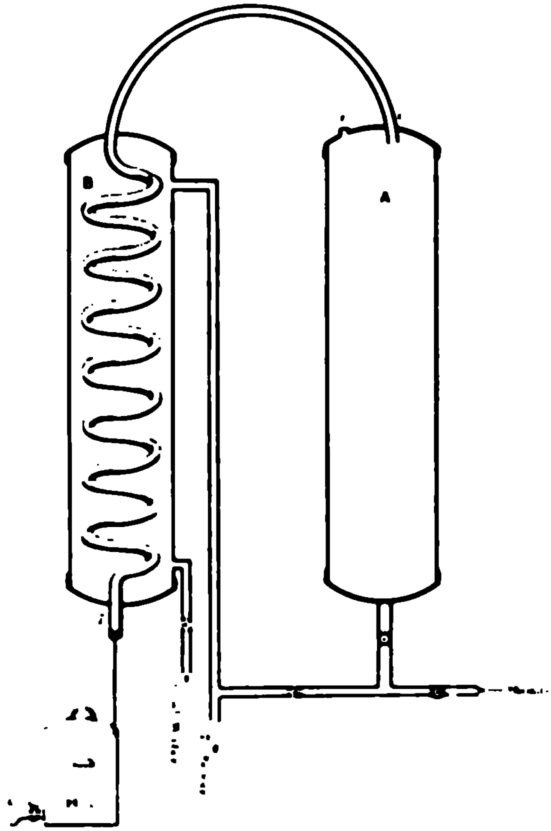
With this modification we can use an asbestos felt and in fodder analysis, after the extraction, it can be taken apart, the substance washed out and used for the crude-fiber determinations, which is a great convenience and saving of time in following the method adopted by the A. O. A. C.

Agrl. College, Md.

DISTILLED WATER APPARATUS.*

By H. J. Patterson

The accompanying illustration shows a distilling apparatus for use with live steam from a boiler. A and B are made of [illegible] inch wrought iron pipe and are 30 inches long; they are fitted with metal heads which screw on very tightly. The pipe C which leads from A, coiling through B to D, is [illegible] inch block tin. A is filled with gravel and serves to hold mechanical impurities of the steam. B is a condenser. E is a [illegible] inch plug for filling with gravel. Steam and water connections are of [illegible] inch pipe.



By means of the arrangement of valves any condensed steam in the pipes may be drawn off before starting it through the filter.

This apparatus [illegible]

[illegible]

[illegible]

*[illegible]

DEVICE FOR ATTACHING BINDING POSTS TO EUDIOMETER TUBES AND OTHER GLASS VESSELS.

By Charles E. Munroe.

Explosion eudiometers, Hofmann's tubes for the electrolysis of water and other substances, Geissler's tubes for showing the effects of electricity in vacuo and similar apparatus as sold in the market are provided with pieces of platinum wire sealed into the walls of the vessel, the ends of the wires which project outside the vessel being looped to receive the conducting wires from the battery or Ruhmkorff coil. As these loops are frequently bent in use it happens that they eventually become broken off and the apparatus is thereby rendered useless. To avoid this I have employed the device shown in Fig. 1, which consists of a split ebonite ring *a* into which a binding post *b* is screwed. This post passes completely through the ring and a thin plate of platinum *c* is soldered to the inner end of it. The device is attached to a tube, as shown in Fig. 2, by means of the set screw *d*. For globular-shaped vessels the ebonite takes the form of a section of the vessel and is attached to it with cement.

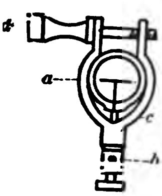


Fig. 1.

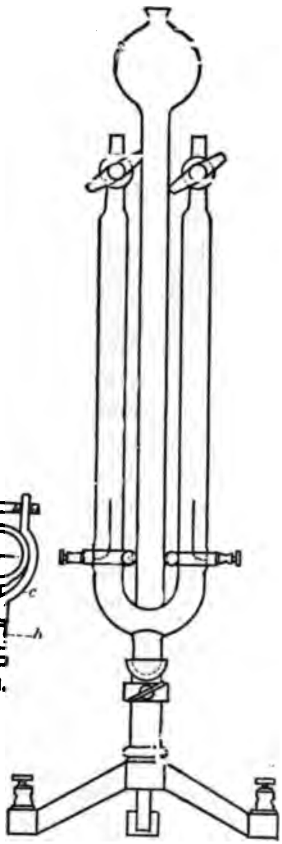


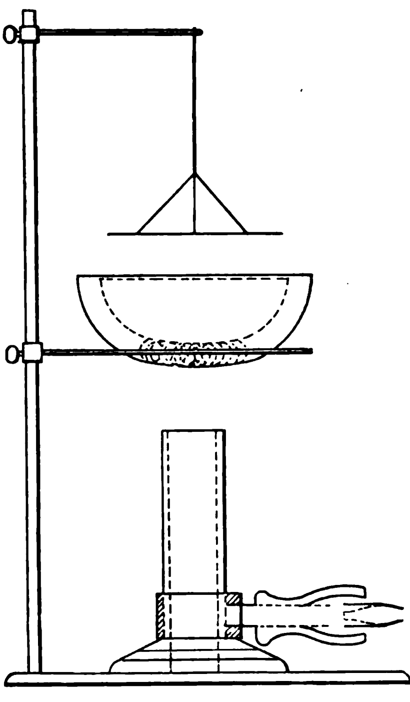
Fig. 2.

Torpedo Station, Newport, R. I.

ON THE "LOSS ON IGNITION" IN WATER ANALYSIS.*

BY THOMAS M. DROWN, M. D.

The loss of weight resulting from the ignition of the total solid residue of evaporation of a water was, before the introduction of modern methods of water analysis, supposed to represent the amount of organic matter present in the water. It is now known that this loss, as ordinarily obtained, by heating the platinum dish containing the residue with the free flame of a Bunsen burner, is a very uncertain quantity. Two chemists will often get widely different results, in consequence of varying practice in the temperature employed and the time of heating.



In order to eliminate this personal equation, as well as to control the temperature of the ignition, I make use of a large platinum dish as a radiator, inside of which the dish containing the residue is heated.

The above sketch shows the arrangement. The radiator is 4⅛ in. in diameter and 2 in. deep. The dish in which the water residue is heated is one inch less in diameter, and rests on a coil of platinum wire, half an inch high. A triangle of platinum wire may be used instead of the coil. A flat disk of platinum foil is suspended one inch over the inner dish, in order to radiate back the heat and hasten the combustion of the organic matter. The radiator is maintained at a moderate red heat by means of an argand gas-burner.

Careful experiments have proved that when solutions of sodium chloride, potassium nitrate and calcium carbonate, either alone or

*Technology Quarterly, Dec., 1888.

in solution together, are evaporated to dryness and heated in this way for half an hour or more, there is no loss in weight. When carbonaceous matter, like sugar, is burned off in the presence of calcium carbonate, the latter loses no carbon dioxide. Organic matter would doubtless decompose nitrates, but the quantity of nitrates present in surface waters is so small that the weight of the fixed solid residue would seldom be affected by their partial or entire decomposition. In fact, nitrates are to some extent reduced to nitrites by the heat of the radiator alone, when no organic matter is present; but the amount of this reduction is generally too slight to be appreciated by the balance.

The following determinations were made on the same sample of filtered Cochituate water, in order to compare the loss on ignition in the radiator with that obtained by cautiously heating the dish to dull redness with the Bunsen burner held in the hand. In both cases the brown residue of evaporation was heated until the carbonaceous matter was completely burned off, leaving a white residue; and after this ignition, the residue was moistened with a dilute solution of ammonium carbonate, dried, and the residue again very gently heated to drive off the excess of ammonium salt.

Amount taken.	Loss in Radiator. (Parts per 100,000.)	Loss over Free Flame.
100 cc.	1.20	2.00
200 cc.	1.10	1.95
200 cc.	1.10	1.75
200 cc.	1.15	1.65
500 cc.	1.10	1.70
1000 cc.	1.17	1.77
1100 cc.	1.18	1.68

These results show that not only is the loss much lower in the radiator, but that it is much more uniform.

It has been found that moistening the ignited residue with ammonium carbonate solution is unnecessary when the ignition is carried out in the radiator, since no carbon dioxide is driven off; moistening with water and drying at 100° C. answers the purpose —restoring any water of combination which may have existed in the residue—quite as well.

The determination of the total solid residue and loss on ignition in ground waters is an entirely different problem from that

presented in surface waters. In uncontaminated ground waters there is little or no organic matter, and the mineral matters, consisting in part, it may be, of lime, magnesia, chlorine, and nitric acid, are generally present in large amount. On the evaporation of a solution of this character to dryness there is loss of chlorine due to the decomposition of the magnesium chloride, and the solid residue is difficult to weigh owing to its deliquescence. On ignition there is loss of water of crystallization and of chlorine and nitric acid ; indeed, the "loss on ignition" under these circumstances bears no relation whatever to organic matter.

This difficulty can be remedied by adding an amount of sodium carbonate solution, of known strength, to the water in the platinum dish before evaporation, sufficient to make it slightly alkaline. The lime and magnesia are then precipitated as carbonates —compounds which are not deliquescent, do not contain any water of crystallization, and are permanent at the temperature to which they are exposed in the radiator. The chlorine and nitric acid combine with the alkali and give compounds which are likewise permanent. When the "loss on ignition" is obtained under these conditions in ground waters, or in the effluents from the purification of sewage by filtration, it will represent approximately the organic matter. In good ground waters the loss is nothing. The amount of sodium carbonate added is, of course, to be deducted from the weight of the total and fixed residue.

One cannot expect the same sharpness of results in determining the solid matters and loss on ignition in water analysis as is obtained in the determination of the nitrogen compounds. If 200 cc. of water are taken for evaporation, an error of weight of one milligram is equivalent to 0.5 part in 100,000. When we consider the number of conditions which may affect the weight of the residue of evaporation, both before and after ignition,—such as the temperature of the air bath, the time of heating, the character of the residue, the hygroscopic condition of the atmosphere, the condition of the air of the laboratory, whether containing much dust, or acid or ammoniacal fumes,—the lack of agreement among chemists in this simple determination is not surprising.

The practice in the chemical department of the Massachusetts Institute of Technology is as follows: Two hundred cubic centi-

metres of a surface water, or one hundred of a ground water which contains considerable mineral contents in solution, are evaporated to dryness on a water bath in a hood which is used for no other purpose. To the ground water is added, if necessary, sodium carbonate, as above described. When the water is all evaporated, the platinum dish is placed in the air bath, connected with the water bath, for about one hour. The temperature of this air bath is 95° to 98° C. The dish is then transferred to a desiccator and cooled over sulphuric acid for half an hour: it matters not how long it stays in the desiccator after it is cool. It is then weighed, and in order that the residue may not absorb moisture during weighing, there are placed in the balance case four beakers of sulphuric acid, which is frequently renewed. This is essential in those cases in which the residue has a tendency to absorb moisture. The dish is next heated in the radiator until all the organic matter is burned off, moistened with water, and put again into the air bath, that it may be, at the second weighing, precisely under the same conditions as the first weighing.

The loss of weight in surface waters, which blacken on ignition, may, under these conditions, be regarded as a close approximation to the organic matter present, both vegetable and animal; and the residue, or "fixed solids" represents, fairly well, the mineral matters of the water.

MASSACHUSETTS INSTITUTE OF TECHNOLOGY,
November, 1888.

A METHOD FOR THE DETERMINATION OF FAT IN MILK.

BY CHARLES L. PARSONS.

Some time ago I tried to make some analyses for fat in milk by the method of B. Röse (Zeit. für angewandte Chemie, 1888, Heft 4, S. 100) using only petroleum ether as a solvent. I was not successful, however, as the two liquids would form no sharp separation, and with one or two individual cows some of the fat was not dissolved. After several experiments I obtained a method which has worked with all of the milks analyzed by it.

8 to 10 gms. of milk are weighed from a small beaker into

a 250 cc. pear-shaped flask. The milk is heated at once to boiling over the flame of a gas or alcohol lamp, bumping and burning of the milk being prevented by continual motion of the flask. As soon as the boiling point is reached, the flask is removed from the flame, 20 cc. of 95% alcohol is at once added, and the whole cooled down by placing in a beaker of cold water. 100 cc. of gasoline is now placed in the flask, which is closed and shaken for one-half minute, after which a little less than 1 cc. of strong NH_4OH is added, and the flask again shaken for the same time. The flask is now allowed to stand quiet for fifteen minutes and then shaken for about a minute. As soon as the gasoline fat solution separates and clears up, 25 cc. of the upper liquid are measured off with a pipette into a small tared flask, the gasoline distilled off and the fat dried at 100° C to constant weight. The amount of fat multiplied by four gives the amount in the milk used; the volume of the fat being so small in comparison to that of the solvent that it need not be taken into consideration except in the analysis of cream. When cream is analyzed the approximate volume of its fat should be considered in the calculation.

The heating of the milk to boiling I have found necessary only in exceptional cases, but it should always be done as a precaution. One milk gave without heating the following results.

Without heating, first	1.37
" " second	3.74
" " third	1.18
Milk heated to boiling	4.58
Gravimetric	4.59

The gasoline employed was that which is ordinarily used in gas machines, and began to boil at about 25° C, the last portions distilling over at about 60°. As a rule, redistillation was unnecessary, as the gasoline left no residue on evaporation.

With this method I have obtained the following results. These analyses were made upon the milk of twenty-three individual cows, of eight different breeds,* each cow of full blood, and upon the mixed milk of several cows.

*The eight different breeds were Durham, Jersey, Ayrshire, Holstein, Devon, Normandy, Guernsey and Swiss.

No.	Milk.	Gravimetric Method.	Gasoline Method.
1	Whole milk of single cow,	4.58	4.64
2	" " " "	4.65	4.66
3	" " " "	4.87	4.78
4	" " " "	3.63	3.70
5	" " " "	3.47	3.58
6	" " " "	3.54	3.65
7	" " " "	4.00	3.98
8	" " " "	3.68	3.79
9	" " " "	4.87	4.91
10	" " " "	5.33	5.44
11	" " " "	4.54	4.63
12	" " " "	4.58	4.64
13	" " " "	3.60	3.72
14	" " " "	3.98	4.07
15	" " " "	3.74	3.72
16	" " " "	3.77	3.70
17	" " " "	3.96	3.88
18	" " " "	3.67	3.75
19	" " " "	3.46	3.42
20	" " " "	3.72	3.75
21	" " " "	3.71	3.66
22	" " " "	3.38	3.33
23	" " " "	3.56	3.68
24	" " " "	4.97	5.02
25	" " " "	4.66	4.74
26	" " " "	4.54	4.59
27	" " " "	3.72	3.68
28	" " " "	4.57	4.64
29	" " " "	3.73	3.64
30	" " " "	3.21	3.16
31	" " " "	2.92	2.94
32	" " " "	5.00	5.05
33	" " " "	3.95	4.00
34	" " " "	3.52	3.57
35	" " " "	4.01	4.10
36	" " " "	5.07	4.99
37	" " " "	5.64	5.70
38	" " " "	4.45	4.58
	Milk evaporated on pure quartz sand for solids; afterward whole shaken with alcohol and 100 cc. gasoline; 25 cc. taken.		
39	" " " "	5.02	5.05 4.95
	Gravimetric on fresh milk; with gasoline, milk 48 hours old.		
40	" " " "	4.59	4.58

No.	Milk	Gravimetric Method	Gasoline Method
	Gravimetric on fresh milk; with gasoline, milk 72 hours old, curdled.		
41	Whole milk of single cow,	3.45	3.26
	Gravimetric on fresh milk; with gasoline, milk 100 hours old, sour.		
42	" " " "	3.48	3.38
	Average	4.11	4.15
43	Mixed milk of several cows .		4.09 4.10
44	" " " " . .		4.91 4.94
45	" " " " . .		3.03 3.17
46	" " " " . .		3.22 3.22
47	" " " " . .		5.15 5.13
48	" " " " . .		3.39 3.41
49	" " " " . .	4.23	4.20 4.14
50	" " " " . .		4.47 4.50
51	" " " " . .		5.80 5.77
52	" " " " . .		5.20 5.12
53	" " " " . .		5.81 5.90
54	" " " " . .	4.91	4.94 4.98
55	" " " " . .		5.54 5.59
56	Skim-milk	.56	.66 .67
57	"	.37	.41 .46
58	"	.14	.18
59	"	.25	.23

No.	Milk	Gravimetric	Gasoline Method
60	Cream	21.66	21.55 21.38
61	"		25.38 25.55
62	"		28.79 28.67
63	Butter-milk	.51	.58
64	"	.30	.34 .32
65	"	.26	.29 .30

The analyses of mixed milk were in series, unchecked by other gravimetric methods, except in a few cases. In each series the milk, skim-milk, butter-milk and cream were analyzed and their total weight taken. The total pure fat found in the butter corresponded closely with the amount found in the whole milk less the amount lost in the skim- and butter-milk.

Inspectors of milk in our large cities still generally use the method of Wanklyn for the estimation of fat in milk. They probably do so for three reasons. In such methods as those of Soxhlet and B. Röse the cost of the large amount of ether used forms no small item where a large number of analyses are to be made, while in Wanklyn's method the cost of the solvent is merely nominal. Wanklyn's method requires nothing but the most simple apparatus; and a larger number of analyses can be finished in a given time than by other gravimetric methods.

I claim for the above method these advantages possessed by Wanklyn's for city laboratories, together with greater accuracy, and hope that in such laboratories it may find application.

MACARONI AND ITS COMPOSITION.

By Clifford Richardson.

Since the production, in this country by the modern roller process of milling wheat, of flour of a character suitable for macaroni, the manufacture of this article of food has been introduced by Italians into Brooklyn and Chicago, where large

amounts are annually made. With a view of comparing the quality and composition of the domestic article with that imported from Italy and other countries, the following samples, collected by Chas. A. Condit, of New York, have been examined and analyzed.

	Place of Manufacture.	Manufacturer.
425	Brooklyn, New York.	Italian Labor
426	" "	" "
427	" "	" "
428	" "	" "
429	Italian	
430	"	
431	Naples	Ricaotti Paturzo
432	Bordeaux	Veuve Garres Jne. et Fils
433	Clermont Ferrand	Marge Goy
434	Lyons	Girord & Hours
435	Lyons	" "
436	Bordeaux	L. A. Price
437	Valence	
438	Gragnans Naples	N. Garofalo
439	" "	"
440	" "	A. N. Cuomo
464	Lyons	Henri Manelline
465	Gragnano Naples,	Alfonso di Nola

	Price per lb.	Size.	Quality.
425	.04	Largest	Second
	.05	"	Fair
	.06	"	Extra
	.07	"	Fair, Dark
135	3rd Ave. New York	Smallest	Solid, Good
430	" " "	Medium	Good
	.10	Largest	Good
	Macaroni Mezzani Extra	Large	Good
	" Moyen "	Medium	Second
	" Taganrok "	"	Extra
435	Afrique 1er Choix	"	Second
	Spaghetti Extra	Smallest	Solid, Extra
	" "		Fair
	Macherone de 1r Qualita, Zita	Largest	Second
	" " Mezzani	Large	Second
440	Extra	"	Good
464	Macaroni Taganrok	"	"
465	Macaroni Best Qual.	"	"

Serial No.	Color	Water	Ash	Oil	Albuminoids	Starch, Fiber, etc., by Difference	Total	Nitrogen of Albuminoids
425	Brown	9.70	1.05	1.25	13.43	74.57	100.00	2.15
426	Lt. Brown	9.45	.79	.75	12.63	76.38	100.00	2.02
427	Lt. Yellow	9.13	.55	.70	11.56	78.06	100.00	1.85
428	Lt. Brown	8.91	.62	1.05	13.31	76.11	100.00	2.13
429	Light	8.34	.95	.85	14.63	75.23	100.00	2.34
430	Lt. Brown	8.93	1.00	.78	11.82	77.57	100.00	1.89
431	Yel'w Brown	9.26	.82	.80	13.13	75.99	100.00	2.10
432	Lt. Brown	8.55	1.12	.78	11.31	78.24	100.00	1.81
433	"	9.28	.74	.70	13.75	75.53	100.00	2.20
434	"	8.29	.73	.91	11.31	78.96	100.00	1.81
435	Brown	8.69	.82	.85	13.37	76.27	100.00	2.14
436	Light	8.53	.74	.70	10.25	79.78	100.00	1.64
437	Very Brown	8.20	.80	.88	13.88	76.24	100.00	2.22
438	Yel'w Brown	7.99	.79	.92	14.63	75.67	100.00	2.34
439	Brown	8.05	.74	.86	13.50	76.85	100.00	2.16
440	"	8.68	.87	.78	13.44	76.23	100.00	2.15
464	Lt. Brown	9.31	.69	.90	13.69	75.41	100.00	2.19
465	"	9.03	.60	.70	10.31	79.36	100.00	1.65

The specimens were first noted as to size and color, and then, after cooking, carefully as to quality, including appearance and taste.

The analysis was carried out according to the methods of the Association of Official Agricultural Chemists for 1887–8, the determination of fiber being omitted as in flour of the grade used for the manufacture of macaroni, which must be the best, the amount is too small to be of importance, not reaching one per cent.

Practically it was found that one of the American samples made in Brooklyn and sold at six cents per pound had the best color and most delicate flavor of any of the macaronis. Its only failing was that owing to its large size and slight deficiency in gluten there was a tendency to lose shape on cooking. The other American specimens were not of the best quality, although one was more expensive than the best sample. This seems to be due to the too stiff or glutinons quality of the flour used in their manufacture as evidenced by the high percentage of albuminoids. In fact in almost all cases the more delicate and better colored macaronis were those not containing high percentages of nitrogen. A deficiency in this element however tends to destroy the proper

consistency and to make the sample too soft and shapeless on cooking, a feature not as noticeable in the smaller spaghetti as it is in the larger kinds, no. 436 a French spaghetti with only 10.25 per cent. of albuminoids being of extra quality and not displaying in its small form the lack of consistency.

It may therefore be stated that a medium percentage of albuminoids is the most desirable feature of a good macaroni, although the darker varieties with large amounts may be more nutritious. In fact the same considerations seem to apply as in the production of white and palatable bread.

It is stated that flours which are too stiff are sometimes reduced with starch, but it is probable that as the darker colored macaronis with high percentages of albuminoids are made from flours known as the "Baker's Grade" this is not always practiced. No. 429 however shows the possibility of making a "good" macaroni with the highest percentage of albuminoids, but the necessity, when compared with the other samples rich in nitrogen, of avoiding anything but the fancy "patent" flours, which are of whiter quality, owing to their lower percentage of oxidizable oil and albuminoids which have a tendency to discolor. From knowledge of the character of our flours derived from previous investigations and comparison with the facts obtained from the examination of the specimens of macaroni in hand there seems to be no reason why the best most palatable and nutritious article should not be made in this country from our high grade Minnesota and Dakota spring wheat roller process flours.

Washington, D. C., March [illegible]

[Contributions from the Laboratory of the U. S. Dept. of Agriculture.]

QUANTITATIVE DETERMINATION OF ADULTERANTS IN LARD.

[illegible]

In a former communication* I spoke of the use of the refractive index in estimating the probable percentage of adulteration in lard and the value of this method when used alone for this pur-

* [illegible]

pose. I now desire to extend the observations made at that time and to include a comparative study of the extent of adulterations when judged by all accessible standards. The methods in use may be classified as follows:

(1). By weight of undissolved residue when the mixed fat is treated with ether.

(2). By the relative intensity of color produced by sulphuric acid and other reagents.

(3). By the relative quantities of silver or gold reduced, or intensity of coloration, in Bechi's, Milliau's and Hirschsohn's processes.

(4). By calculation from specific gravity.

(5). By calculation from iodine absorption.

(6). By calculation from refractive index.

(7). By determination of the insoluble matter produced by treatment with chloride of sulphur.

(8). By rise of temperature with sulphuric acid.

(9). By calculation from the melting point.

(10). By the coloration produced by heating with nitric acid and albumen. (Brullè's method.)

In the peculiar conditions attending the analysis of mixed lards it is unnecessary to say that the most misleading results may be obtained by relying on any one of the above methods, and even when all are applied the real quantity of added adulterants may not be determined. The process indicated in Nos. 1, 2, and 3 of the foregoing classification may be dismissed without further discussion. They are unreliable for any quantitative purpose. In the case of No. 4 approximate results could be reached were only one kind of adulterant used, the specific gravity of which, as in the case of cotton oil, is distinctly different from that of lard.

But if one adulterant be used like an oleo or lard stearine having a lower specific gravity, and another like cotton oil with a higher one, the neutralizing effect of the two will render the results of the analysis unreliable.

Cotton oil, however, has a specific gravity considerably higher than that of a stearine is below the number for pure lard; hence, a mixed lard containing equal portions of cotton oil and a stearine will have a higher specific gravity than pure lard. In point of fact it may be said that where one of these adulterants is present in any notable quantity, say 15 to 30 per cent., the other is also present in proportions approximately known. It might be possible, therefore, to construct an arbitrary formula by which the disturbing effect of the second element could be allowed for. In this way some approximate number might be reached of the respective amounts of adulterant present.

Example:

Let specific gravity of pure lard	at 35°	=.905
" " " " " stearine	"	=.903
" " " " " cotton oil	"	=.913

The theoretical specific gravity of a mixed lard composed of these bodies in the proportions stated would be as follows:

20%	stearine	=.903×20=18.060
30%	cotton oil	=.913×30=27.390
50%	lard	=.905×50=45.250
100%		=90.700

Then theoretical specific gravity=.907

It is usual to mix cotton oil and stearine in compound lards in the respective proportions mentioned above, viz., 1.5 to 1.*

The specific gravity of the mixture is therefore

Cotton oil	1.5 parts=.913×1.5	=1.3695
Stearine	1. part =.903×1	= .9030
Mixture	2.5 parts =	2.2725

Theoretical specific gravity=.909

The following table therefore will give the approximate percentage of adulterations corresponding to the specific gravities noted:

*Testimony of George H. Webster, Report of Hearings, p. 26, before Senate and House Committees on Agriculture.

Table Showing Approximate Percentage of Adulteration Corresponding to Different Specific Gravities when the Adulterants are Cotton Oil and Stearine in Respective Proportions of 1.5 to 1.

Observed Sp. Gr. at 35°	Percentage of Pure Lard.	Percentage of Adulterant.	Percentage of Cotton Oil.	Percentage of Stearine.
.9050	100.00	00.00	00.00	00.00
.9055	87.50	12.50	7.50	5.00
.9060	75.00	25.00	15.00	10.00
.9065	62.50	37.50	22.50	15.00
.9070	50.00	50.00	30.00	20.00
.9075	37.50	62.50	31.50	25.00
.9080	25.00	75.00	45.00	30.00
.9085	12.50	87.50	52.50	35.00
.9090	00.00	100.00	60.00	40.00

A general expression for the calculations above when applied to other standards of temperature and actual results obtained may be easily devised. The general formula, however, will still rest on the assumption that the cotton oil and stearine are mixed in the proportions noted, and this will be found to be practically the case.

Let s=the observed specific gravity at T°
a=the specific gravity of pure lard at T°
Let b=the specific gravity of pure cotton oil at T°
c=the specific gravity of pure stearine at T°

$\frac{1.5\,b+c}{2.5}$=the specific gravity of the mixed adulterants at T°

Let x=per cent. of adulteration.

$$\text{then } x=\frac{100\,(s-a)}{\frac{1.5\,b+c}{2.5}-a}$$

For illustration, we may apply this formula to the analytical data collected. Each analyst should carefully determine for himself, in a great number of samples, the true specific gravities of the various substances entering into the mixture at the temperature used by him as a standard.

Example:

Mean specific gravity of pure lard at 35°	.9053=a
" " " " cotton oil "	.9142=b
" " " " stearine "	.9015=c

" " " " the cotton oil and stearine mixed (calculated) . . $.9091=\frac{1.5\ b+c}{2.5}$

The mean specific gravity of Armour's lard, was .906.

$$\text{then } x=\frac{100\ (.906-.9053)}{.9091-.9053}$$

whence $x=18.42$ per cent.

The mean specific gravity of Fairbank's lards was .9095. This shows a theoretical adulteration of over 100 per cent., or in other words, a lard composed wholly of stearines and cotton oil, in which the oil is in slightly greater proportions than those indicated above. The iodine number obtained shows that the lard approximates such a composition.

(5). *By Calculation from Iodine Absorption.*

The determination of the percentage of iodine absorbed by a mixed lard taken alone, cannot lead to any just idea of the amount of adulterant added.

In the case of specific gravities the numbers for oleo-stearine and lard-stearine are near together, viz., for 35°, .900 and .902 respectively. But for iodine numbers the difference is very great. In the three samples of oleo-stearine examined the mean iodine number is 20.73 per cent. In the two samples of lard-stearine analyzed it is 47.02 per cent. The mean number for cotton oils is 109.02 per cent; for lard 62.48 per cent, and for prime steam lard 62.86 per cent. In a mixture we may find all of these ingredients, and therefore the iodine number of such a mixture may approximate that of a pure lard.

When the iodine number of a supposed adulterated lard goes above 65 per cent., there are grave reasons for suspecting an adulteration with cotton oil, but a pure lard made from certain parts of the hog may show even a higher number.

If the microscopic examination shows the presence of oleo-

stearine and cotton oil be revealed by the silver or gold tests, the complexity of the problem is less confusing.

The iodine number may then reveal the approximate quantities of the adulterant present. For example :

1.5 parts of cotton oil at 109	=	163.5
1 part of oleo-stearine at 20	=	20.0
2.5 parts	=	183.5
1 part	=	73.4 per cent

Now a mixed lard whose iodine equivalent is about 64 per cent. (Armour's) cannot be made of any considerable quantity of the above mixture and pure lard. It must contain a notable quantity of lard-stearine. For example :

40 parts of cotton oil and oleo-stearine	at 74	=2960
30 parts of lard	at 62	=1860
30 parts of lard-stearine	at 47	=1410
100 parts		=6230

The theoretical iodine number of such a compound lard is therefore 62.30 per cent. The above hypothetical example in the light of the analysis made, shows approximately the composition of a compound lard whose iodine number is not above 63 per cent.

In the Fairbank samples the mean iodine number is 85.31 per cent. The microscope revealed also the presence of oleo-stearine in these samples. They were presumably composed of cotton oil, lard- and oleo-stearines, and perhaps some lard. As was shown by the specific gravity they contained an excess of cotton oil.

These mixtures may be represented by the following proportions :

10 parts oleo-stearine	at 20	= 200
25 parts lard-stearine	at 47	=1175
65 parts cotton oil	at 109	=7085
100 parts		=8460

theoretical iodine number=84.60 per cent.

No formula can be given for computing the proportions of in-

gredients from the quantity of iodine absorbed, except in the tentative way indicated above; but the value of the iodine number, when thus studied with other quantitative data, is sufficiently illustrated.

(6). *By Calculation from the Refractive Index.*

Some valuable information concerning the quantitative composition of a mixed lard may be derived from a study of the refractive index. The mean refractive index at 25° of the samples of lard examined is 1.4620; water at the same temperature showing 1.3300.

For cotton oil the number is 1.4674
For oleo-stearine the number is 1.4582
For lard-stearine the number is 1.4594

The determination of a much larger number of samples of the stearines would be desirable before deciding on a permanent standard, but the above numbers will serve provisionally.

The difference between lard and cotton oil is	+54	points
" " " " oleo-stearine is	—38	"
" " " " lard-stearine is	—24	"

It thus appears that the addition of cotton oil to a lard would raise its refractive index, while the addition of the stearines would lower it. In general, it appears that two parts of stearine would neutralize the effect of one part cotton oil. A mixture of 1.5 parts of cotton oil and 1 part of mixed stearines would have the following theoretical index:

1.5 parts cotton oil at	1.4674	=2.2011
1 part stearines at	1.4588	=1.4588
2.5 parts		=3.6599
1 part		=1.4640

For a lard adulterated with the above mixed adulterant we may use the following formula:

Let r=observed index at 25°
a= " " of lard.
b= " " " cotton oil.
c= " " " stearine.

$$\frac{1.5\,b+c}{2.5}=\text{index at }25^\circ\text{ of the mixed cotton oil and stearine.}$$

Let x=percentage of adulteration.

$$\text{then } x=\frac{100\,(r-a)}{\dfrac{1.5\,b+c}{2.5}-a}$$

As an illustration of this formula take the mean numbers obtained in the tables of samples for lard, cotton oil, stearines and Armour's mixtures.

Observed	index	of Armour's samples	$r=1.4634$
"	"	" pure lards	$a=1.4620$
"	"	" cotton oils	$b=1.4674$
"	"	" stearines	$c=1.4588$

then $x=.14\div.0020=70$ per cent.

According to this formula, Armour's samples would have only 30 per cent. of pure lard, a result which is contradicted by other data. I am inclined to believe that the examination of a larger number of samples of stearines may show a higher index, and thus bring the results obtained by the application of the above formula more into harmony with the other data. The index for the Fairbank samples, 1.4651, shows that in these mixtures cotton oil has been used in greater proportions than indicated above, thus corroborating the results obtained by the other methods of analysis. Judged by the index of refraction alone, on the assumption that this index for the stearines is not much different from that of lard, the composition of a mixed lard is probably as truly indicated as by any other single method.

(7). *By Determination of Product formed by Chloride of Sulphur.*

Warren has obtained some interesting results, and our own work has shown that much may be expected of a careful study of this process. Lack of time has prevented a full investigation, and this will be made subsequently.

(8). *Rise of Temperature with Sulphuric Acid.*

Valuable information relating to the composition of a mixed lard may be obtained by a study of rise of temperature of a given volume thereof when mixed with a definite quantity of strong sulphuric acid. The data obtained in our analyses are as follows:

Rise of temperature with	lard	41°.5
" " " "	cotton oil	85°.4
" " " "	oleo-stearine	20°.8
" " " "	lard-stearine	37°.7
Mean " " " "	the stearines	29°.3

When the microscope reveals oleo-stearine we may take the last number to represent the mean increment of temperature. For an adulterant composed of 1.5 parts of cotton oil and 1 part of stearine the mean rise of temperature would be 63°. The apparent composition of a mixed lard on the above character of the adulterant would be illustrated by the following formula:

Let t = observed rise of temperature for sample
a = " " " " " lard
b = " " " " " cotton oil
c = " " " " " stearine

$$\frac{1.5\,b+c}{2.5} = \text{theoretical rise of temperature for the adulterant.}$$

Let x = percentage of adulteration

$$\text{then } x = \frac{100\,(t-a)}{\dfrac{1.5\,b+c}{2.5} - a}$$

This formula applied to the mean rise of temperature observed in Armour's samples gives the following result:

x = 23.3 per cent.

Applied to Fairbank's samples, it shows an adulteration of 76.3 per cent.

(9). *Melting Point.*

The melting point of a fat is often of great value in helping to a correct understanding of its composition; but little reliance can

be placed on it for quantitative purposes. The different glycerides when mixed do not have a melting point which corresponds to the one theoretically calculated. For this reason equal mixtures of cotton seed oil and lard instead of having a melting point of about 20°, really melt at a much higher temperature. While, therefore, the determination of the melting point of a compound lard should not be omitted, it does not afford a basis for any reliable estimation of a quantitative measure.

(10). The coloration produced by heating the fat or oil under examination with nitric acid and albumen has also been proposed as a quantitative test. Although I have not tried this method quantitatively, I am of the opinion that it will be found of no greater value than the other color reactions already noted.

The Brullè test appears to be unaffected by free acid or rancidity, in which point it possesses an advantage for qualitative purposes over chloride of gold, and in some cases over nitrate of silver.

Summary.

From the methods already worked out, as applied to the two classes of mixed lards examined, the following general results are deducible, viz.:

Method of Examination.	Sample from	Per cent. of Adulteration.
By specific gravity	Armour & Co.	18.42
" refractive index	"	70.00
" rise of temperature	"	23.30
" specific gravity	Fairbank & Co.	100.00
" refractive index	"	100.00
" rise of temperature	"	76.30

The mean per centage of adulteration for the Armour samples is 37.24.

For the Fairbank samples it is 92.10.

It is not unusual to omit the percentage of lard-stearine used in accounts given by manufacturers of the extent of adulteration. If we allow that one-third of the total adulterant is lard-stearine the percentages of foreign fats in the Armour and Fairbank lards are 24.83 and 61.40 respectively.

In the foregoing discussion it has been assumed that the mean properties of a mixture of various glycerides are proportional to the quantities of each present. In the case of the melting point we know that this is not the case, and the consideration of the melting point therefore as a factor in quantitative determination has been omitted.

It may be true that other properties are also unequally developed in a mixture, and this would add still another complication to the problem.

In the present state of our knowledge the chemist is unable to express definitely the degree of adulteration which a sample of lard has suffered. He can state with confidence whether or not a given sample is adulterated, and in the comparison of two widely different sets of samples, such as were obtained from Armour & Co. and Fairbank & Co., he may safely say that one is adulterated to a greater degree than another. Further than this the present state of our knowledge will not permit us to go.

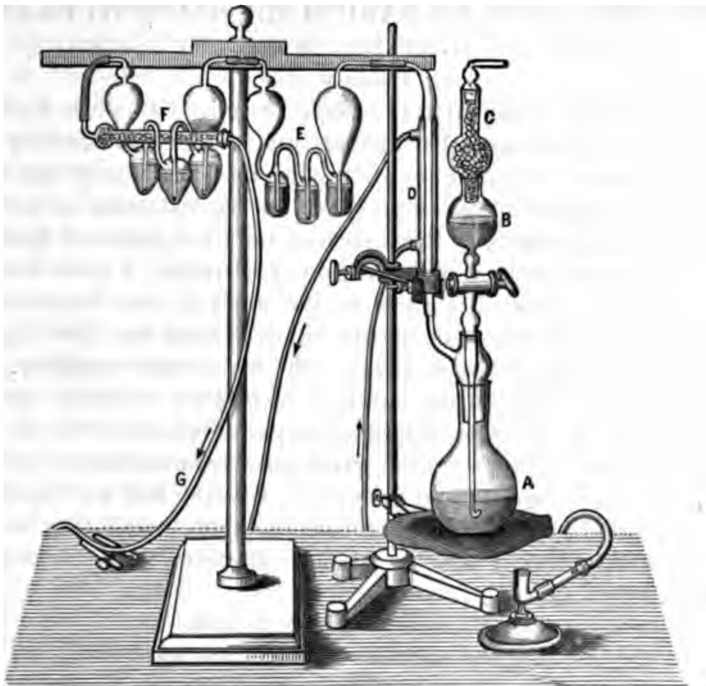
A MODIFIED APPARATUS FOR DETERMINING CARBONIC ACID.

BY AUG. E. KNORR, U. S. DEPT. OF AGRICULTURE.

An apology perhaps should be offered for adding another apparatus of this kind to the number which is already legion. However, the modification here described has filled an apparent want, and has given entire satisfaction in this laboratory.

The advantages claimed for it are that it is permanently mounted, and that the danger of a possible leakage through defective rubber joints is reduced to a minimum.

The apparatus proper, as represented by the cut, consists of a flask *A* in which the carbon dioxide is set free. A condenser *D* is ground into the neck of this flask and condenses the steam formed when the liquid in *A* is boiled in order to secure complete expulsion of the gas. The reservoir *B* contains the acid required for the operation, and has a soda-lime guard *C* ground into it to retain the carbonic acid of the air, a constant current of which is aspirated through *G* during the whole operation. The stem of the reservoir is ground into the condenser, or it may be conven-

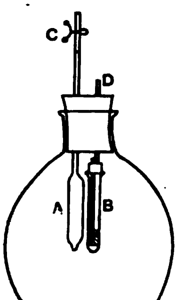


iently blown into one piece with it. The carbonic acid is dried in *E* and finally absorbed in the weighed potash bulb *F*.

A NEW CARBONIC ACID APPARATUS.

By E. R. Flint.

The accompanying sketch shows a new form of carbonic acid apparatus devised by W. H. Beal and myself, which for compactness and light weight, has advantages over any other form that has come under my notice. The acid holder (A) and the desiccator (B) being both inside the flask, makes the apparatus much more convenient to weigh than the majority of its kind. The acid is dropped in by opening the pinchcock (C) through which opening the desiccated air enters when aspirating, and passes through the long tube in the test-tube, through a little sulphuric acid and thence passing out at (D), thus insuring a thorough circulation through the flask.



Mass. State Ag'l Exp't Station, Amherst.

THE REDUCTION OF BARIUM SULPHATE TO BARIUM SULPHIDE ON IGNITION WITH FILTER PAPER.

By Charles W. Marsh.

In reading on the subject of some barium salts while looking over the preparation of barium sulphide by mean of igniting the mineral barite with coal and extracting the barium sulphide with water, my attention was again called to the reduction of barium sulphate precipitates by the carbon of the filter paper on ignition in the regular method for estimation of sulphur. I have noticed in some text books on quantitative analysis the direction to separate the precipitate of barium sulphate from the filter, ignite the filter, and burn to ash, moisten the mass with sulphuric acid to transform any barium sulphide formed by reduction of the carbon of the filter to sulphate, drive off excess of acid, and ignite again. The precipitate previously separated is now added and the whole ignited and weighed. I could find no literature on the reduction of barium sulphate by ignition with the filter in the determination of sulphur and have therefore made the experiments herein described.

Two series of experiments were conducted.

In the first case the method of procedure was as follows: 25 cc. concentrated sulphuric acid were diluted to 500 cc. and it was with this acid that all the experiments were made. The diluted sulphuric acid was taken instead of a soluble salt to avoid as much as possible liability of error from incomplete washing. For the first ten determinations various amounts of the acid were further diluted with distilled water, heated to boiling and barium chloride solution added, and allowed to stand several hours or over night. The precipitates of barium sulphate were repeatedly washed with water by decantation and on the filter until the filtrates gave no reaction for hydrochloric acid. After drying the precipitates the filters were folded over, enveloping them, and cautiously ignited in a platinum crucible with the lid on until the escaping gases were ignited, when the burner was removed. When the flame went out the burner was again applied and the crucible brought to a red heat. The cover was removed, the crucible placed in a horizontal position and heated until the filter was burned to an ash, cooled and weighed.

A little water was then added to the contents of the crucible

and the whole well stirred, and allowed to settle and the liquid decanted on a filter. In many cases hydrogen sulphide was detected after the addition of water by its odor, and by turning lead acetate paper brown. After having extracted two or three times with water, two cc. of dilute hydrochloric acid were added in small portions with addition of water, whereupon in most cases the evolution of hydrogen sulphide was still more pronounced. The precipitate was further treated with water. If there was an odor of hydrogen sulphide in the filtrate a few drops of bromine water were added and the solution boiled to expel the excess of bromine. A little of the diluted sulphuric acid was added, allowed to stand several hours and the precipitate of barium sulphate collected on a filter, washed, dried, ignited, moistened with concentrated sulphuric acid, and the barium sulphate ignited and weighed.

This precipitate was derived from the barium sulphide formed by reduction of the barium sulphate by the carbon of the filter-paper during ignition, and was dissolved by the water and dilute hydrochloric acid added, and reprecipitated by sulphuric acid.

The total washings varied between fifteen and twenty cc.

The following table shows the resuits obtained by working in the manner described:

	A.	B.	C.	D.	E.
1.	*.9844	.0068	.0048	.9864	.67
2.	1.0291	.0073	.0053	1.0311	.70
3.	.9926†	.0377	.0273	1.003	3.75
4.	1.0287†	.025	.0181	1.0356	2.41
5.	.9634*	.0071	.0051	.9654	.73
6.	1.1462†	.0915	.0663	1.1714	7.81
7.	1.532†	.0483	.0350	1.5453	3.12
8.	1.9413†	.0679	.0492	1.96	3.46
9.	1.788*	.0127	.0092	1.7915	7.08
10.	1.9247	.1503	.109	1.966	7.64
11.	.2166	.0021	.0015	.2172	.96
12.	.1526	.0012	.0008	.153	.78
13.	.076†	.0147	.0106	.0801	18.32
14.	.1621†	.0039	.0028	.1632	2.38
15.	.0919†	.006	.0043	.0936	6.41

Explanation.—

evolution on addition of acid.

†Odor of hydrogen sulphide on addition of water, with more evolution on addition of acid.
*Odor of hydrogen sulphide when acid was added.

A=weight of $BaSO_4$ and BaS after ignition in the crucible.

B=weight of $BaSO_4$ obtained by dissolving out BaS and precipitating the barium as sulphate.

C=the weight of $BaSO_4$ found in B, calculated to BaS from which it was obtained.

D=the weight of A if it were all $BaSO_4$, obtained by subtracting the weight calculated in C from the weight of A, and adding to the remainder the weight given at B.

E=percentage of $BaSO_4$ reduced, found by dividing B by D and multiplying by 100.

Schleicher & Schülls ashless filter paper, number 590, was used.

In the first ten experiments the diameter of the filter was 9 cm., and in the last five 5½ cm.

The barium sulphate used on the last five experiments as well as in the second series was prepared in one beaker and carefully washed. This saved the individual washings for several precipitates at the same time giving a precipitate identical in all cases.

The next set of experiments was made in similar manner with this change in manipulation. The ignition of the precipitate was performed without the lid on the crucible, thus giving complete access of air, and more nearly approaching the ignition of barium sulphate as carried out in the determination of sulphur. The results are as follows:

	A.	B.	C.	D.	E.
16.	.1389	not reduced			
17.	.0631	" "			
18.	.061	" "			
19.	.0547†	.0015	.0010	.0552	2.71
20.	.0513†	.0024	.0017	.0520	4.61
21.	.0654	.0003	.002	.0655	4.59
22.	.0514	.0006	.004	.0516	1.12
23.	.0545†	.0011	.0008	.0548	2.00
24.	.0585†	.0012	.0008	.0589	2.04
25.	.0598*	.0057	.0041	.0614	9.28
26.	.7423†	.0069	.005	.7442	.92
27.	.7437†	.0059	.0042	.7454	.79
28.	.6776†	.0122	.0088	.681	1.79
29.	.5042†	.0142	.0103	.5081	2.79
30.	.77†	.0143	.0103	.774	1.84

In the above experiments a filter of 5½ cm. diameter was used in each case but the last five, where one of 9 cm. diameter was used. The average reduction in the first table was 4.41 per cent. of the barium sulphate originally present and in the second 2.29 per cent., a trifle more than one half of that in the first case.

A blank experiment was made on .8582 grams barium sulphate with the same amount of hydrochloric acid and water and but three tenths of a milligram of barium sulphate were obtained. I have not included this in connection with the above table as it is so small.

The before mentioned experiments prove conclusively that there is quite an appreciable amount of barium sulphate reduced by the filter paper in ignition and that the reduction is about twice as much when the ignition is made with the cover on the crucible, as when made without it.

The moistening of the ignited precipitate of barium sulphate with sulphuric acid and again treating should not be omitted in determination of sulphur as already proved.

LEHIGH UNIVERSITY, SOUTH BETHLEHEM, PA.

ABSTRACTS.

APPARATUS AND REAGENTS.

Burette for Titrating Heated Liquids.—L. L. de Koninck Ztschr. angew. Chem. 1888, **187.** The burette is fastened in iron clamps fastened to one side of an iron stand. The evaporating dish in which the titration is to be carried out is supported by a ring fastened upon the opposite side of the same stand and at a lower level. A bent tube passes from the lower end of the burette over the evaporating dish, so that the liquid from the former may be run into the dish while the solution in it is boiled by a lamp placed below.

The Oxidation of Sulphurous Acid and Sulphites.—I. A. Bachman, Am. Chem. J. **10,** 40. Bachman has investigated the oxidation of solutions of the sulphites of sodium, potassium and

ammonium and of acid ammonium sulphite and sulphurous acid. He finds:

First.—There is very little difference in the rate of oxidation of the sulphites of sodium, potassium and ammonium.

Second.—There is a marked advantage in employing an acid sulphite where it may be substituted for a solution of a sulphite or of sulphurous acid, and is to be kept as a reagent or dispensed as a medicine.

A Shaking Apparatus is described by A. Stutzer (Ztschr angew. Chem. 1888, 698). This was designed for determining the amount of fine powder in Thomas slag, but may be used for other purposes. It consists of a stout table supporting a rectangular box. This box supports another of smaller size which moves back and forth on strips fastened inside it. The movable box contains the powder to be sifted placed in sieves which fasten in it and are provided with proper covers. The sifted powder falls through into the lower box. Motion is obtained by means of an eccentric and rod attached to a shaft placed across one end of the table and driven by a crank.

Apparatus for Weighing upon Tared Filters.—L. L. de Koninck (Ztschr angew. Chem. 1888, 690). This consists of a light funnel with short neck ground to fit into a wider tube. This wider tube passes through the cork of a side-neck flask; the side neck is attached to a Bunsen pump. After filtration is finished, the funnel is fitted over a short glass tube which is ground to fit in it and terminates below in a tripod which supports it while drying and weighing; the funnel is covered with a ground glass plate while being weighed. Instead of the ordinary shape the funnel may have the form of a cylinder drawn together above and below and covered with a ground cap; this is useful especially for filtrations through asbestos. [This apparatus seems to be a very poor substitute for the Gooch crucible.]

A Safety Pinch Cock for cutting off the supply of gas when the flame "snaps back" in a Bunsen burner is described by N. von Klobukow (Ztschr. anal. Chem. **27**, 168). We must refer to the original paper for the detailed drawings, without which the apparatus cannot well be described.

Sublimation Apparatus.—J. W. Bruhl (Ber. **22**, 238). This consists of a round hollow box made of nickel-plated brass, with openings on each side for the passage of cold water. In the center of the box is a perforation having the shape in section of a truncated cone. In this opening a crucible containing the substance to be sublimed is placed and is heated from below. The substance sublimes and condenses on a watch glass placed over it.

Improvement in Standardizing Permanganate.—R. Jahoda Ztschr. angew. Chem. 1889, 87. The author keeps an atmosphere of carbonic acid in the flask while the iron to be titrated is dissolving, by connecting the flask with a beaker one-half filled with a solution of sodium bicarbonate. A crystal of the bicarbonate is put in the flask with the iron to drive out the air. Heat is used to aid the solution of the iron, and on cooling the sodium bicarbonate solution is drawn over into the flask, only a few drops, however, as the gas driven off drives the liquid out of the tube. The amount of water that carries over with the bicarbonate can be regulated by the concentration of the soda solution.

J. E. W.

Some New Laboratory Apparatus.—A. Beutell, Ztschr. augew. Chem. 1889, 6.

1. *Drying oven.*—The design is for an oven 84 cm. high, 72 cm. wide and 47 cm. deep. The walls and top are double, the outside being of sheet iron covered with asbestos, the inside of sheet copper. A space is left between the two sheets of metal for the passage of the air, which is heated by four horizontal burners under the oven. The heat is not allowed to strike directly against the bottom of the oven, but is directed to the hollow walls, and a hollow partition which divides the oven into two chambers by two curved tin shields.

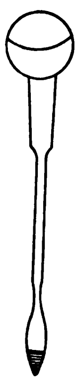
Four thermometers are placed, horizontally, through the sides of the oven, two near the top and the other two a few inches from the bottom. Cold air is allowed to enter the oven through small holes in the sides just below the lower thermometers. By this arrangement the entire air inside the oven may be left at the same temperature.

The passages through the sides and partition convey the hot

air and burned gases to the chimney. The author recommends his oven for all temperatures between 70° and 200°.

2. *Ignition Stand.*—A rectangular framework of heavy iron wire, under which is arranged a series of eight burners; the air and gas are regulated at the same time. The burners are provided with telescope tubes, so that the inner cone of the flame can be brought up against the crucible, which is held on a platinum triangle laid across the framework, the advantage being the economy of room.

3. *Swimmer for Burette.*—The author finds that with a Mohr swimmer there is a difference in the amounts of water drawn from the burette, as the swimmer rises through the liquid, or rises to the 0 point. This is due, he says, to the large surface of the Mohr swimmer, causing great capillarity. He proposes a form of swimmer as shown in the figure. The mean of nine weighings of 10 cc. each of water drawn from a burette, using the Mohr swimmer sinking to the 0 mark, was 10.046 gms. to 10.0329 gms.; with the new swimmer the mean of nine weighings of 10 cc. each of water, using the Mohr swimmer rising to the 0 point, was 9.979 gms. to 10.030 gms. with the new design. In all the experiments, the temperature of the water was practically the same. J. E. W.



Apparatus for Fractional Distillation in Vacuum.—S. B. Newberry (Am. Chem. J. **10**, 362). The distilling flask is provided with a thermometer and separatory funnel; the end of the separating funnel is drawn out to a fine point and reaches to the bottom of the flask. By opening the stop-cock slightly a very slow current of air may thus be drawn through the liquid which serves to prevent bumping and has little effect upon the pressure. The end of the condenser tube fits into the neck of another separatory funnel by a tightly fitting cork; and the stem of the separatory funnel passes through the cork of a flask from which the air may be exhausted by connecting it with the filter pump. In order to change the receiver the cocks of both separatory funnels are closed, the receiver changed and then both cocks again turned to their original positions. In this way it is possible to carry on the distillation as conveniently as under ordinary pressure.

Vapor Density Determination Under Diminished Pressure.—An apparatus for this purpose has been devised by C. Schall (Ber. **22,** 140). We must refer the reader to the original paper. The apparatus used is a modification of that of Victor Meyer.

A Method of Vapor Density Determination.—Theo. W. Richards, Chem. News **59,** 87. The author has described the apparatus he uses in a previous number of the Chem. News (**59,** 39), and gives further details of the method with fuller directions for working it. A. H. W.

The Examination of Reagents.—L. L. de Koninck, Ztschr. angew. Chem. 1889, 4.

Peroxide of Lead.—This salt often contains manganese, sometimes as peroxide, and by itself cannot be tested for by dilute nitric acid. The test in the wet way is made by completely decomposing the oxide with concentrated sulphuric acid, with the aid of heat, allowing the solution to cool, then adding some more of the oxide, and on warming this, if manganese is present, the red color will appear.

Ether.—All commercial ether contains impurities, such as organic sulphur compounds, which on the evaporation of the ether are left as a strong smelling residue. To test for free sulphur, shake a quantity of the ether with a small amount of metallic mercury. If sulphur is present, the mercury will be covered with a gray film of sulphide, and often the whole liquid will be colored.

Chloroform.—To test for alcohol in chloroform, dissolve permanganate of potash in a saturated solution of barium hydroxide and add this solution to the chloroform. If a trace of alcohol is present, the green color of the manganate will show instantly.

J. E. W.

Note on Kjeldahl's Method for Nitrogen Determination.—Meldola and Moritz, J. Soc. Chem. Ind. **7,** 63. The authors call attention to the fact that the sulphuric acid used for this process should be free from nitrogen compounds, and especially from ammonia, which is often added in the process of manufac-

ture as ammonium sulphate. Such acid may be purified by adding a small amount of potassium nitrite and heating the acid when the following reaction occurs:

$$NH_3+HNO_2=N_2+2H_2O.$$

About 0.05 gm. potassium nitrite is sufficient for 10 cc. acid. [This seems to me a very doubtful improvement. Pure sulphuric acid is not expensive enough to make this plan worth while, and the danger of introducing nitrates is obvious.—E. H.]

An Improved Soxhlet-Szombathi Extraction Apparatus. — F. M. Horn, Ztschr. angew. Chem. 1889, 33.

The use of corks in joining together the different parts of an extraction apparatus is a serious objection, as the liquids commonly used are solvents for the gum and wax contained in the corks.

The author has designed an apparatus in which ground joints take the place of corks. Fig. 1 represents this apparatus, which is made about 70 cm. in length. Fig. 2 shows a form of this extractor, in which a double rolled linen bag is used to hold the material. The bag is fastened to the end of the condensing tube just above the flange *c* by a platinum wire. The liquid used is heated in

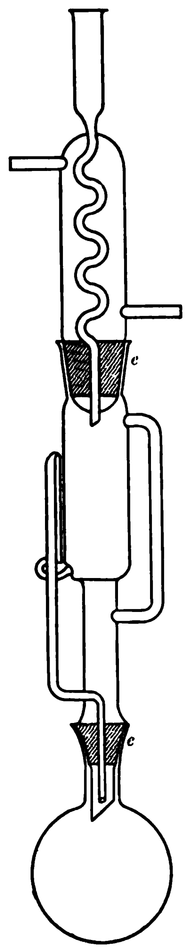


Fig. 1.

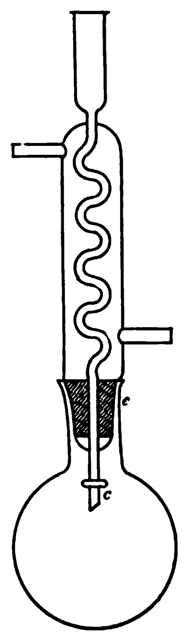
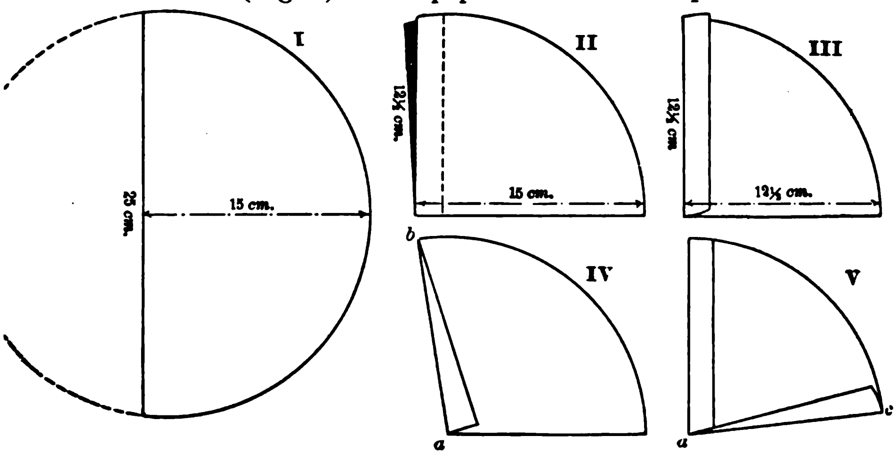


Fig 2.

the flask, the vapor passes through the bag, is condensed in the cool tube, and drops into the material in the bag. The author uses this form with alkaline solutions, water, very dilute sulphuric acid, alcohol or ether. J. E. W.

Improvement in Filter Papers.—Edo. Claassen Amer. Drug. March, 1889. "Patterns corresponding with the size of the filters wanted are cut out of sheet metal and rather less than one-half cut off (Fig. 1). The paper cut to this shape is then



folded as in 2, and the edges rurned over as in 3. This piece when opened out will fit a funnel with an angle of 60°. If the funnel has a greater angle the paper is folded as in 4, and if a less angle, as in 5.

Valve for Use with the Filter Pump.—H. Schaumann, Ztschr. anal. Chem. **27**, 636. The tube from the flask into which the filtrate runs is thrust through a small calcium chloride tube. Over the end projecting through the cork a thick-walled rubber tube, stopped at the farther end, is thrust. A slit is cut lengthwise in the rubber tube. This is Bunsen's valve applied in another way.

Drying and Weighing Glass for Paper Filters.—C. Reinhardt, Ztschr. angew. Chem. 1889, 61.

The apparatus consists of a funnel-shaped glass vessel with a moveable cover. The paper is placed as in an ordinary funnel,

and the apparatus weighed before and after filtration. The author claims the advantage of the conical shape of the paper while the precipitate is drying, preventing its curling up, and also presenting a large surface for drying. The weight of the apparatus is 45 gms. and the largest paper suitable being 12.5 cm. in diameter.

J. E. W.

Extraction Apparatus and Water Bath.—P. N. Raikow, Chem. Zeit. **13**, 94.

The author shows a convenient form of apparatus for extracting substances with cold ether in the following cut, Fig. 3.

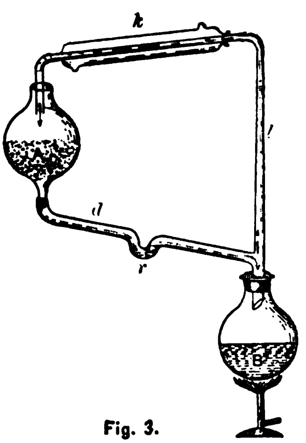


Fig. 3.

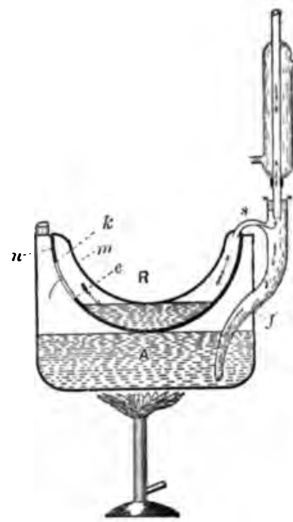


Fig. 4.

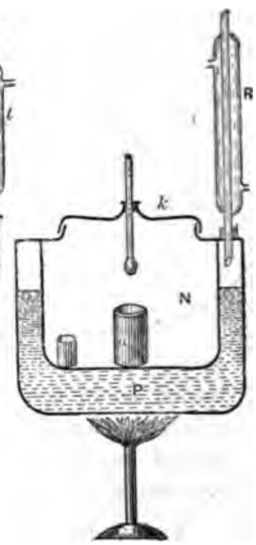


Fig. 5.

The ether is placed in the flask *B* and heated. The vapors are condensed by the condenser *K* and pass through the material to be extracted *A* and return to the flask *B*. The trap *r* prevents the hot vapor from reaching *A*.

The water bath is a continuous one, and is shown in Fig. 4. It consists of an ordinary copper vessel provided with a double cover, the upper one *m* being air tight and the lower one *n* having a slit *k e* about 2 mm. wide cut in one side for the passage of water between the covers. These covers are 5 or 6 mm. apart.

The steam passes between the covers into the condenser *t* and the water from there runs back to *A*.

A drying bath on the same principle is shown in Fig. 5.

S. C.

Apparatus for Determining Carbonic Acid.—W. Hanks, Chem. Zeit. **13,** 358. The author modifies the old form of apparatus, in which the carbonic acid is determined by taking the weight of the apparatus before and after the carbon dioxide is liberated by another acid, by fastening to the calcium chloride tube a tube filled with pumice stone saturated with copper sulphate solution to prevent any loss of hydrochloric acid.

S. C.

Measuring Glass for Fractional Distillation.—Hofelmann, Chem. Zeit. **13,** 357. This consists of a glass tube with a bulb blown in the middle and the lower end mounted on a wooden base. The tube is divided into 10 cc. below the bulb and the graduations run from 80 to 100 cc. above the bulb. S. C.

Apparatus for Crystallization in a Freezing Mixture.—J. W. Brühl (Ber. **22,** 236). Made from a wide mouthed bottle by breaking off the bottom; this when inverted and closed with a cork serves to hold the freezing mixture. The cork has two perforations, one for a tube with pinch cock to let out the freezing mixture as it liquifies, the other for the passage of the stem of the inner vessel; the inner vessel which contains the liquid to be frozen is made of a large pipette which is broken off just below the upper contraction; this is fitted with a perforated cover which fits over it by a ground joint. The perforated cover is fitted with a cork, in which is inserted one end of a drying tube. The lower end of the pipette is provided, in the contraction, with a perforated platinum funnel for retaining the crystals which form. The stem of the pipette passes through the cork of the broken-off bottle and is joined to a tube having a glass stop cock, which in turn passes through the cork of a side-necked flask connected with the Bunsen pump.

The Practical Value of Thomson's Calorimeter.—S. Kestner Dingler's poly. J. **271,** 171. The calorimeter of Lewis

Thompson consists of a copper cylinder closed below, to contain a mixture of the combustible substance, (especially coal,) potassium nitrate and chlorate. Four springs are fastened to the base of this, which hold it in place in a large copper cylinder, open at the bottom and provided above with a tube and stopcock. A fuse fastened in the mixture in the inner cylinder is ignited, the outer cylinder slipped over it, and the whole apparatus immersed in a beaker of water. When the combustion takes place the gases pass out under the edge of the outer cylinder and give up their heat to the water in bubbling through it. The cock is then opened, to allow the water to enter the combustion chamber, after which the whole apparatus is moved up and down in the beaker, acting as a stirrer, and the temperature taken. From the weight of substance used, the weight of water and the rise in temperature the amount of heat liberated can be calculated.

The results given by this apparatus have now been compared by Kestner with those of the Favre and Silbermann Calorimeter. The result of his work is that the error of Thompson's calorimeter does not often exceed four per cent., and that it often gives results much closer than this; so that, for approximate results where rapid work is important, it answers very well.

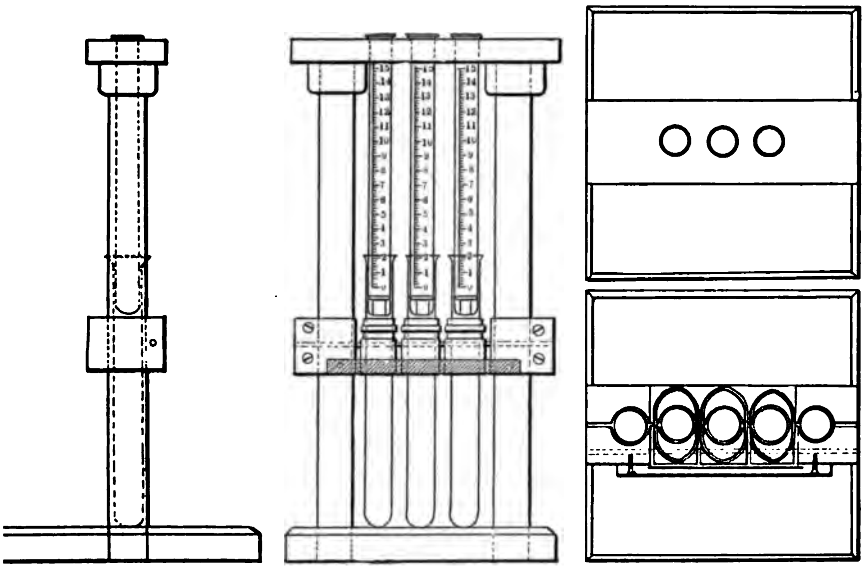
A Simplified Chromometer.—Especially adapted to the estimation of carbon in steel is described by C. H. Risdale (J. Soc. Chem. Ind. **7**, 70) as follows:

"*Description of Apparatus.*—Three glass comparison tubes of equal bore throughout, graduated to 15 main divisions, each of which is sub-divided into fifths, hang as near each other as possible through a perforated strip of wood. This, in turn, is supported at each end by a thick glass rod, terminating at its lower extremity in the base board. The centre tube, which can be used for the standard, is sealed at the bottom, whilst the two side tubes have a $\frac{3}{16}$th hole in them. In all three is a small enamel glass cylinder, the top of which is the zero line, and the graduation continues upwards.

Clamped on the two glass rods which support the top piece is a grip arrangement, holding three ordinary test tubes, which slide easily over the graduated comparison tubes, and the tops of

which, when in their lowest position, are about up to the zero mark of the graduated tubes. The grip arrangement consists of three fixed jaws, against which are held, by india rubber bands, three moveable jaws pivoted on a horizontal axis, the jaws being hollowed so as to grip the test tube sliding in them. They serve to keep the sliding tube at any height to which it may be raised.

"*The process of Comparison* is as follows:—Fill the centre tube up to 10 divisions (or any other number preferred) with solution of standard depth of tint, pour the solution to be compared into



one of the side tubes, when it will at once run through the hole in the bottom into the test tube beneath, and when this is nearly full, look right down the two comparison tubes and raise the test tube beneath, containing the solution to be compared (keeping the hand below the grip all the while so that it does not cast a shadow on the liquid in the tubes), until the depth of tint is equal to that of the standard.

"Then read off the number of divisions required, and calculate:

$$\frac{\text{Number div. stan.} \times \text{per cent. stan.}}{\text{Number div. sol. to be tested}} = \text{per cent. sought.}$$

Then pull out the comparison tube and the test tube, rinse and return to their places, put in the next solution to be tested, and so on.

"The centre sliding tube should be raised to the same height as the side one, so as to equalize any effect which the double thickness of glass may have upon the tint.

"It is of course a matter of indifference whether a measured quantity of *standard* solution be put into the centre tube, and the height of the column of solution of unknown strength adjusted till it is the same tint, or a measured quantity of the *unknown* solution put in the centre tube, and the standard solution adjusted: indeed where the solutions to be tested vary a good deal in tint I have used a perforated tube for the centre also, and adjusted *both* colums, using fewer divisions of standard for the weaker and more for the deeper tints, this being, of course, on account of the comparatively small number of divisions on the tubes.

"If a measured quantity of the *solution to be tested*, say 10 divisions, be taken, and the height of the column of *solution of known strength* (the standard) adjusted, then if the standard contains .10 per cent. of the body being estimated, the percentage of it contained in the sample being tested can be read off *direct* from the scale.

"For instance, if we are estimating carbon in soft steel, with a standard containing .10 per cent. of this body, and we find that 10 divisions solution of unknown strength require 8 divisions solution of standard, then the sample being tested contains .08 per cent. C.

"If 12 divisions solution of standard were required, the sample would contain .12 per cent. C., each division being equal to .01 per cent.

"The same of course applies to the estimation of copper in steel, pig-iron, ferromanganese, &c., to ammonia in water by Nessler's test, or to any other colorimetric estimations, to all of which it is equally applicable.

"In short, to read the percentage straight off, all that is necessary is to use as many divisions of the solution of unknown strength as there are hundredths per cent. of the body to be

estimated in the standard. Thus, if our standard steel contained .12 per cent. C. we should use 12 divisions of the solution of unknown strength.

"If the hundredths per cent. of constituent in the standard be some number higher than the graduations of the apparatus show, as many divisions solution of unknown strength can be used as will when multiplied by 2, 3, 4, &c. give the hundredths per cent. contained, *e.g.*, in testing rail steel we use a standard with .3 or .4 per cent. C., and still take 10 divisions solution of unknown strength and multiply the divisions standard required by 3 or 4. In cases where the particular class of work done renders it desirable, the graduations could of course be altered to suit requirements.

"It will be seen that there is *no* pumping of solutions as in the other chromometer*, and indeed nothing that can get out of order.

"I have used this apparatus chiefly for testing carbon in steel by the 'Eggertz' color method, and proceed as follows: Weigh off .1 to .2 grams drillings into a test tube 6 inches by $\frac{5}{16}$ th, add dilute nitric acid 1.20° sp. gr. quantity as shown below, put into boiling water, shaking the tube every minute or so till the steel is dissolved, then make up with cold water to the bulk indicated below.

Weigh off.	For Steel containing probably carbon. Per Cent.	Dissolve in cc. HNO_3 1.20° Sp. Gr.	Make up with cold water to a bulk of cc.
.1 or .2 gms.	Up to .08	1¼	10
		2½	20
.1 gm.	.08 to .19	1½	20
.1 ,,	.20 to .30	1¾	40
.1 ,,	.31 to .40	2	80

"Compare these solutions with those of standards of about the same content treated similarly. To facilitate the operation of 'making up' for ordinary soft steel up to .19 per cent. C., test tubes can be used which have a file mark to show how high to fill them for 10 and 20 cc. They then only need to be shaken, and the solution poured into the apparatus. If all of the same gauge they could, if prefered, be simply inserted in the grip, but

* See this Journal I, 221.

it is questionable whether this saves any time, and it only restricts the use of ordinary test tubes. One advantage of using this apparatus for comparison instead of the ordinary method of adding water drop by drop to the solution of unknown strength till it is sufficiently diluted is, that in the latter case too much water may be added, thus giving too high a result, and if this is done the extent to which it has been over diluted can only be guessed, and as every drop may make a difference of .01 to .02 per cent., the result is often seriously invalidated, whereas in the apparatus the tints can be adjusted until they are absolutely the same, the readings taken, and then anyone else can check the comparison. Further, all the solutions can be easily kept till it is seen whether any of the results appear doubtful.

"Again, steels dissolved in dilute nitric acid usually show a greenish yellow tint, due to the nitrate of iron, but which, when diluted considerably, disappears. This tint interferes very much with the ordinary 'Eggertz' method when the sample being tested contains under .1 per cent. C., and it becomes practically impossible to distinguish the amount of carbon with any degree of certainty by that method when the percentage is below .07.

"Our younger assistants almost always fail to discriminate between it and a real carbon tint, and in consequence add too much water, so that if they come unexpectedly upon a sample containing .08 per cent. they probably show the result too high.

"With the apparatus, however, the nitrate of iron tint is destroyed because the solutions are diluted to an extent which would be inadmissible by the ordinary modification of Eggertz' method.

"A large number of samples containing from .07 to .09 per cent. carbon showed this amount *when done very carefully* by the ordinary method, *making due allowance for the nitrate of iron tint*, and these results also agree with those given by the apparatus, but when tested by junior assistants showed from .02 to .03 per cent. higher, being over diluted to that extent. Indeed so completely is the difficulty hitherto caused by the nitrate of iron tint overcome, that the carbon in even *extra* soft steel can be very fairly estimated by this apparatus.

"The following samples, all of which by the ordinary Eggertz

method showed .05 to .10 per cent. C. when tested as above, gave the appended results:

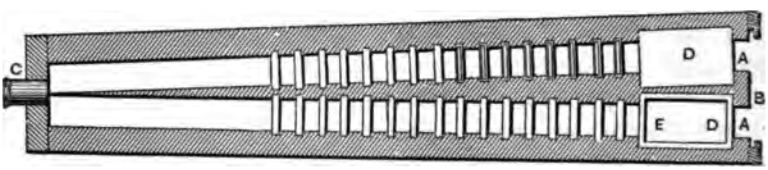
No.	C. content by Alkali Method and Rotary Chromometer. Per Cent.	C. by Method described and new Deep Tint Chromometer. Per Cent.
1	.042	.043
2	.040	.040
3	.037	.041
4	.035	.041
5	.045	.046
6	.040	.042
7	.040	.040
8	.020	.021

"Thus it will be seen that though perhaps not quite as accurate for minute traces of carbon as the ordinary rotary chromometer in conjunction with Stead's alkali method, yet it yields results which are near enough for most purposes, thus showing approximately right results when samples containing a very low percentage of carbon are come upon unexpectedly, and where the ordinary Eggertz method would fail entirely.

"As regards *rapidity*, when the apparatus is clean it takes no longer to make the comparison this way than by the ordinary Eggertz method, and when doing a quantity there is the advantage that one assistant can make up to the required quantity and another test them, so that they can be got out in remarkably little time. The making up and testing by the *same* person of the eight samples for which results are just given only took 12 minutes, of which time the greater part was spent in making up and shaking, the comparison only taking two or three minutes. The total time from putting on to dissolve to the finishing of the last result was 17 minutes, whereas the time by the alkali method and rotary chromometer would have been for the eight samples 40 to 45 minutes.

"In the matter of *accuracy*, besides the examples already cited I have checked the results repeatedly against standards which have been analysed by several chemists by the ordinary Eggertz color method, Stead's alkali method, and by combustion in oxygen, and have found it thoroughly reliable, and a great assistance in obtaining accurate results rapidly."

The Tintometer.—J. W. Lovibond, J. Soc. Chem. Ind. **7**, 424. This "consists of a tube divided by a central taper partition B terminating in a knife edge at the eye-piece C. This knife edge, being inside the range of vision, is not seen when the instrument is in use. At the other end of the instrument are two apertures D D, of equal value, alterable in size and shape by means of diaphragms. The two apertures are here divided by the thick end of the central partition B, which, together with the sides, is recessed by grooves in order to hide the edges of the standard glasses and of the vessels placed in the tube for observation. The top is provided with slots in line with the grooves, to admit and guide the standard glasses, and the whole is



arranged in such a manner that the only light which can reach the eye must pass in equal quantities on each side of the partition, up the two tubes, illuminating the object under examination on one side and the measuring glasses on the other. A reference to the diagram * * * will show the arrangement.

"For measuring color in opaque objects the instrument is fitted to a hinged stand, capable of being placed at such an angle as reflects the light from the whitened bottom and sides through the tubes to the eye, so that on looking through the eye-piece two white equal fields of view are seen. The object to be measured is placed on the stage under one tube, and the standard glasses worked in the other, against the white background.

"The instrument as now shown is the sum-total of attempts to overcome difficulties which were discovered and met in detail whilst carrying on color investigation of a commercial character, and until remedied made the work unreliable and misleading. The causes of error were traced to many and various sources: to the intensity of light from opposite directions morning and evening, to the lights and shadows cast by large objects both outside and inside the room, to the window or apparatus not being cen-

tral, to the bars of the window, and even to reflection from the section of wall outside the window. Also, until tubes were discarded in favour of parallel-sided vessels, the convexity of the tubes, which magnified these errors as by a lens, caused disappointment, after apparently obtaining a perfect balance of color, to find that a small alteration of position entirely upset the most careful reading.

THE STANDARD GLASSES

"Consist of colored slips cut to a size fitting the grooves of the instrument. The slips are arranged in series, all the slips of each series are of the same color, varying only in depth of color; the variation is in regular degrees, each degree being of the same tint-value as that chosen for the starting point or unit of the series. Thus a single glass of 10 degrees tint-value has the same value as 10 glasses, each of one degree. Every slip is marked with the number of degrees or the tint-number which it represents, and also with a denominational or *color-number* showing to which series the slip belongs.

"The tint formed by the superposition of several glasses belonging to the same color series is equal to the aggregate of tint-numbers on the glasses used, whilst the superposition of glasses from several series gives a compound color which follows the laws applicable to the mixture of pigments, in contra-distinction to the laws belonging to the mixture of spectrum colors.

THE UNIT.

"In fixing the value of the unit the first effort made was to find a basis of common value applicable to all colors, but the unequal absorption of light by different colors proved an effectual barrier, some building into darkness by superposition of glasses at a much quicker rate than others, thus, with a medium north daylight a red requires 77 of its own units, a blue 222, and a yellow 239. The differences are still greater in more composite colors.

"From this it was evident that each color having its individual rate of absorption, it must have its individual progressive unit, which, with a few exceptions, is not applicable to another color.

"This enforced limitation of the value of each unit to its own color is, as regards a common basical unit, restrictive, but beyond that point very expansive, permitting the unlimited addition of new colors for standards without in any way altering the value of those already in use, or interfering with the value of registered work previously done. It was ultimately decided that the eye being the sole judge of difference, and noting that the powers of differentiation decreased as the tint increased in depth, the tint unit should be as light as consistent with the possibility of the addition of a single unit being distinguishable in the deeper shades; while for lighter shades, where the perception is keener, the tint unit may be divided, if necessary, into decimals of the original unit. The range of color which can thus be brought to measurement is very wide, ranging from most of the brilliant aniline colors to the dingiest cloth, the only apparent limit lies in the variety of colored glass obtainable for standards.

MEASUREMENT OF LUMINOUS BODIES.

"Adopting Professor Church's definition of all visible bodies as being either luminous or illuminated, I propose first to deal with luminous bodies in reference to their measurement for intensity and colour.

"*For Intensity.*—To measure for intensity, having closed one tube by a stop, it is necessary to pass the rays to be measured through the other tube, under definite conditions as to distance, area, screen, or other governing influences, then intersecting these rays with graded glasses of a neutral tint, until the light is entirely blotted out, when the number of degrees required to accomplish this can be read off and will be an index of intensity.

"*For Color.*—Circumstances have prevented much attention being as yet given to the color of illuminants, and I can do little more than suggest some few precautions which would probably occur to any investigator. Rays from the illuminant should be caught on a screen commanded by one tube under known conditions, as already described in measuring for intensity, whilst the other tube should command a standard white field of view against which to work the measuring glasses.

"One of the instruments on the table is roughly arranged to

show the spectrum nature of the coloured light from red, blue, and green glass reflected from a whitened surface, and passed up one of the tubes, the glasses themselves being kept out of sight, as against the pigmentary nature of colours obtained when similar glasses are superimposed in the other tube; the use of graded glasses gives a new interest to this class of investigation.

MEASUREMENT OF ILLUMINATED BODIES.

"*Transparent.*—For measuring and recording tint and color in transparent liquids, a graduated series of accurately gauged vessels with parallel colorless glass ends are adapted to the instrument. These range in thickness from $\frac{1}{16}$ of an inch to two feet, the smaller vessels being suitable for the darker liquids, whilst the blue color in distilled water can be easily measured in two feet. Care must, however, be given both in the distillation and to the pure whiteness of the background.

"When the color of a liquid represents a property or substance the graded glasses of different lengths and the color-scale afford a ready means of quantitative estimation of extreme nicety and ease of management, carrying the accuracy of the balance beyond weighable quantities down to the smallest color trace, as well as being available for larger percentages. The method has been applied to the estimation of color in dyes, Nessler's ammonia test, carbon in steel, copper both as nitrate and ferrocyanide. It is also under examination for lead as sulphide; here the presence of iron would be a disturbing element. There must be many such possible applications, which would often effect a saving of time and labor by avoiding the necessity of evaporation, drying, and weighing, as well as estimating percentages too small for the balance.

"The conditions to be complied with are, first the color must bear a definite relation to the substance producing it, and must be in most cases matchable by the glasses of a single color series. Then by means of a solution of known strength in one of the gauged glasses, the percentage of substance represented by the tint of a single unit must be found by direct experiment. In estimating an unknown sample of a similar substance, it would therefore be theoretically only necessary to use the same gauged glass, and multiply the units required to match it by the percen-

tage value of a single unit; but this cannot always be done, as the progressive variations in the color properties of different substances required great care before finally deciding on the percentage value of a single tint-unit, for the law of regular progression does not always hold, and percentage estimations by means of color can only be made within this law. The great majority of colored solutions will, however, at some point of dilution, give the necessary conditions for a quantitative estimation.

"In making preparations for color testing by methods now in use, such as Eggertz' carbon test, and Nessler's ammonia test, the apparatus should be specially useful as a check upon all possible errors which may arise in preparation, and by bringing the practice of all laboratories to a common standard of reference. It is also found useful in noting and recording changes of color and tint caused by chemical action, time, or other cause.

"*Opaque Objects.*—In measuring color in opaque objects the instrument is fitted to a hinged stand capable of being placed at such an angle as reflects the light through the tubes from a stage on which is placed the object under examination, and a pressed plaster of Paris reflector as a standard white to measure from, or the substance to be compared or matched."

A New Burette Float.—Nic. Wolff (Chem. Zeit. **13**, 389) claims that a paraffine disc 2 mm. thick and about 2 mm. less in diameter than the burette makes a float which is easier to read from than the one commonly used. S. C.

Apparatus for Fractional Distillation under Diminished Pressure.—Gust. Kolbe (Chem. Zeit. **13**, 389.) This embodies the same principles as the one already described.* S. C.

INORGANIC ANALYSIS.

A Gasometric Method of Determining Nitric Acid.†—Percy F. Frankland (J. Chem. Soc. **105**, 364) determines nitrous acid in the following manner: The substance containing nitrous acid is mixed with a large excess of urea crystals, a small

*This Journal **1**, 394.
†See this Journal **2**, 188.

amount of water is added to dissolve the nitrite and urea, and the solution transferred to a small graduated tube fitted with a stop-cock, by allowing the solution to enter the tube as the mercury which has previously filled the tube is allowed to run out. The solution enters from a cup at the upper end of the tube. Dilute sulphuric acid (1 in 5) is added and the contents vigorously shaken. Carbon dioxide and nitrogen are evolved. The former is absorbed by sodium hydrate solution and the remaining nitrogen measured and its weight calculated.

This method compared with the estimation with permanganate of potassium and the method with mercury and sulphuric acid gave concordant results.

The addition of two drops of strong potassium hydrate solution prevented the loss of nitrous acid on evaporation with a peptone.

From the experiments described the author points out that nitrous and nitric acids may be estimated in presence of organic matter in the same sample, nitrous acid by means of the urea method and nitric acid by the mercury method, having previously evaporated the sample with ammonium chloride to destroy nitrous acid. C. W. M.

Note on the Determination of the Free Caustic Alkali in Soap.—J. A. Wilson, Chem. News **59**, 40. When soaps made by the so-called cold process are dissolved in alcohol to estimate the free alkali by titration with decinormal acid and phenol-phthalein, the free acid saponifies the free glycerides, and such soaps would not show free caustic alkali after their solution, although they had done so before. The author's method is to estimate the alkali in total by titration with normal acid and methyl orange, and in a separate portion, the alkali required to neutralize the fatty acids separated from the soap. A. H. W.

Analysis of Commercial Sodium Sulphate.—Isbert and Venator, Ztschr. angew. Chem. 1889, 66.

About 2 gms. of the material is dissolved in the smallest possible amount of water, ammonia and then ammonium carbonate added until no further precipitate forms; aftering filtering, the precipitate is dissolved in dilute nitric acid, again precipitated, and washed with hot water. The precipitate contains the insoluble

matter, the iron oxide and alumina, and the lime and magnesia as carbonates. The filtrates contain all the sodium sulphate and chloride and the free acids. Evaporate the combined filtrates in a platinum dish, ignite gently to expel ammonia salts, and weigh.

The chloride of sodium is estimated, with silver solution, in a separate portion, calculated as sodium sulphate and deducted from the total amount in the dish, the difference is the amount of sodium sulphate in the sample. J. E. W.

Ash Determinations.—E. Kassner, Chem. Zeit. Rep. **13**, 37. The author hastens the combustion of graphite, coke and similar substances by mixing with them finely powdered silver and gradually heating to redness, first in a closed and finally in an open crucible. The silver acts as a carrier of oxygen, and may be recovered at the end of the operation. Chemically pure silver powder may be obtained by adding silver chloride to an alkaline grape sugar solution or by reducing it with iron or zinc in an acid solution. S. C.

The Determination of Ash.—F. A. Fluckiger, Ztschr. anal. Chem. **27**, 637. Substances which burn slowly under ordinary conditions, such as albumen, horn, etc., may be burned quickly if they are first ignited gently, then moistened with water, dried and again cautiously ignited. This is much simpler than Reese's method*. A method which involves the same principle has already been described in these pages†.

The Use of Hydroxylamine Hydrochlorate in Quantitative Analysis.—A. Lainer, Monatshefte f. Chemie **9**, 533. Hydroxylamine hydrochlorate when added to silver nitrate solutions precipitates the silver as chloride. If caustic soda or potassa be then added and the liquid heated the silver chloride is decomposed, silver separating. The hydroxylamine acts upon silver bromide and and iodide in an alkaline mixture in a similar manner. If the hydroxylamine be added to an alkaline solution of silver in sodium thiosulphate the silver is completely thrown down in the metallic form. It is more difficult to separate all the silver from an alkaline potassium cyanide solution, but the last

*This Journal.
†Ibid **2**, 208.

traces may be thrown down by evaporation to a small bulk with repeated addition of hydroxylamine hydrochlorate. Care must be taken in this case to avoid loss by frothing, which is very energetic upon the first addition of the hydroxylamine. The results given show that the method is a good one. It will be especially useful in examining photographic residues and plating solutions.

A New Method of Chemical Analysis in which Capillary Attraction Plays an Important Part.—Charles W. Phillips, Chem. News 58, 285, and Pharmaceutical Record, Nov 1, 1888.

The author invented his scheme in 1872, but was unable to perfect all the details until recently. He first prepares test-papers as follows: Chemically pure filtering paper, with a close grain, is immersed in a ten per cent. solution of ferrocyanide of potassium, laid on a pane of glass, and dried in the open air, or better, in a drying oven at 150° F. Ten per cent. solutions of neutral potassium chromate, potassium bromide, and sodium hyposulphite are also made and paper prepared from each in a similar manner. The test-papers are now ruled off into small squares and each one marked with the symbol of the metal intended to be applied to it. The papers are placed on a clean pane of glass, and not removed until the tests have been applied and are perfectly dry. A piece of glass tubing 7 inches long and 3-16 of an inch inside diameter is drawn to a point, which is then filed off and the end is held in a flame and rotated until a perfectly round end is formed, with an aperture of about 1-50 inch. The tube is dipped into the solution to be tested, say potassium sulphate, a little of the liquid is drawn up, and the tube is placed vertically upon the potassium square on each one of the four test-papers. Using solutions of all the metallic elements in like manner a set of tables for verification may be made. A. H. W.

New Method for the Determination of Water in Silicates.—P. Jannasch (Ber. **22**, 221). The mineral is very finely ground, mixed with a large amount of very fine lead chromate and ignited in a glass combustion tube of 10–12 mm. diameter and 46–47 cm. long, exclusive of the bayonet which should have a length of 12 cm. The filling of the tube is conducted with all the precautions

for excluding water ordinarily observed in making an organic analysis. The method of filling the tube is shown below.

Air Stream. →	Cu Spiral.	Coarse Chromate.	Fine Chromate.	Mixture with the Substance	Fine Chro-mate.	Granular Chromate.	Cu Spiral.
	1	10	1.5	6.5–7	5	18	1–2 cm.

In this way the carbon dioxide can be determined at the same time as the water. That part of the tube containing the mineral must be heated as strongly as possible for at least ¼–½ hour.

Determination of Nitrogen in Nitrates by Kjeldahl's Method.—Otto Foerster, (Chem. Zeit. **13**, 229). The author gives in detail the method already mentioned in this Journal.* 0.5 gm. of the nitrate is heated with phenol-sulphuric acid in the distilling flask and evaporated to dryness. The residue is dissolved in 6% phenol-sulphuric acid and to this solution are added 1 to 2 gms. of sodium thiosulphate, 10 cc. sulphuric acid, and the usual amount of mercury. During these operations the flask must be kept cool. After the action has ceased, the contents of the flask are heated until the organic matter is destroyed, which usually requires 1½ hours. A phenol-sulphuric acid containing more than 7% or less than 4% phenol injures the results. The absorption apparatus is similar to a nitrogen bulb having a capacity of about 200 cc. S. C.

The Rapid and Sure Detection of Antimony in Minerals.—Alexander Johnstone, Chem. News **59**, 15. When but a very small quantity of charcoal is present, the charcoal method must be used (See this Journal **3**, 80), but in other cases, the way recommended is as follows: A mixture of the powdered mineral and a flux is put into a glass tube (¼ inch bore) at a distance of one-quarter of an inch from the end. The tube is then slightly inclined and heated with the blowpipe and the white sublimate which forms on the sides of the tube is tested with a single drop of ammonium sulphide. A. H. W.

*Vol. **3**, p. 73.

The Physical Properties of the Graphites Obtained from Various Metals after the Separation of the Same by Means of Dilute Acids.—H. N. Warren, Chem. News **59**, 29.

Graphite is obtained most readily from metals of the third group by heating them at a high temperature in contact with carbon. Iron gives the largest scales when thus treated, while the graphite from nickel and cobalt is much finer, and with the naked eye cannot be distinguished from lamp black. Ferro-manganese gives a brown graphite, much less readily oxidized and much thinner than that from iron, while the commercial black oxide gives also brown scales, some decidedly translucent and less combustible than those from ferro-manganese. Chrome iron gives much brighter and harder scales than in the case of iron, while cast chromium dissolved, produces scales without any brown tinge, of semi-metallic lustre, not readily oxidized, unless heated in a current of pure oxygen, and approaching nearly to silicon. Difference in the fusion points of the different metals is suggested as a cause for this variation in the properties of the graphites. A. H. W.

Note on the Determination of Copper by the Iodide Process.—Rowland Williams, Chem. News, **58**, 272. 63 grains of the sample were dissolved in the theoretical amount of dilute nitric acid, nitrous fumes were then driven off by boiling, sodium carbonate was added until a slight permanent precipitate formed, then acetic acid in slight excess, and the solution was diluted up to 10,000 grains and one tenth (6.3 grains of the sample) was measured out for each titration. Potassium iodide in excess was now added, and after standing a few minutes, decinormal hyposulphite solution was used for titration. The author finds the method admirable for pure copper solutions, but impurities, lead, iron and arsenic in particular destroy its utility. Both ferric acetate and ferric nitrate liberate iodine from potassium iodide and arsenic acid also decomposes the iodide. A. H. W.

Separation of Zinc and Nickel.—H. Baubigny, Chem. News **59**, 88; Comptes Rendus, **108**, 236. The mixed salts of zinc and nickel are dissolved in water containing 100 cc. of glacial acetic acid per liter, sulphuretted hydrogen is passed through the liquid

at the ordinary temperature, and the solution is allowed to stand two or three hours and then filtered. The zinc precipitate, which ought to be perfectly white, is washed with water containing sulphuretted hydrogen, 10 per cent of acetic acid, and the nickel is precipitated from the filtrate, as customary, the temperature being raised to 70°–75°, and then the solution is allowed to cool in the presence of sulphuretted hydrogen in a stoppered flask. The free acid must be acetic, ammonium acetate being added to secure this end. The sulphides are converted into sulphates and weighed after drying at 400°. If the mixed sulphates contain little nickel, one-third at most, the acetic acid is useless. A. H. W.

The Action of Sulphuretted Hydrogen upon Arsenic Acid.—L. W. McCay (Am. Chem. J. **10**, 459.) According to Wackenroder,* Ludwig† and H. Rose,‡ sulphuretted hydrogen first reduces arsenic acid to arsenious acid with precipitation of sulphur and then precipitates the arsenic as the trisulphide. Fuchs confirmed this view. Bunsen§ showed that under certain conditions the arsenic was precipitated as pentasulphide from an acid solution. The author has shown¶ that all the arsenic may be completely precipitated as pentasulphide from an acid solution by causing the sulphuretted hydrogen to act upon the arsenic acid under pressure and in absence of air. According to Fresenius,** the pentasulphide is obtained only when the solution is kept at 70°. Brauner and Tomicek [illegible] find that only with a hot solution and rapid current of gas is the pentasulphide alone formed. The author finds that when a slow current of sulphuretted hydrogen is passed through an acid solution of arsenic heated to 70° until the solution begins to show an opalescence and then the solution quickly poured upon crushed ice a solution is obtained which after blowing air through it to drive off excess of sulphuretted hydrogen, gives the following reactions:

1. When rendered strongly acid with sulphuric or hydrochloric

[illegible] 97.
[illegible] 107.
[illegible] 8.
[illegible] 191.
[illegible] 1.
[illegible]
[illegible] 8. [illegible]

acid it remains for some time clear. 2. Mixed with sulphuretted hydrogen it gives no precipitate. 3. After boiling and shaking vigorously it becomes opalescent and sulphur is deposited. 4. After the boiled liquid has cooled and the liquid has been separated with asbestos an addition of sulphuretted hydrogen gives a precipitate of arsenic trisulphide. 5. Sulphate of copper produces no precipitate. 6. Mercuric chloride gives a heavy yellowish white precipitate. 7. Silver nitrate gives a heavy black precipitate of silver sulphide. These reactions are produced by sulphoxy-arsenic acid,* H_3AsO_3S, the acid potassium salt of which has been described by Boquet and Cloez.† When this acid in solution is heated it decomposes, forming sulphur and arsenious acid.

The action of sulphuretted hydrogen upon arsenic acid in acid solution may therefore be expressed by the following reactions :

(1). $H_3AsO_4+H_2S=H_3AsO_3S+H_2O$

(2). $2H_3AsO_3S+3H_2S=As_2S_5+6H_2O$

(3). $2H_3AsO_3S+6HCl=As_2Cl_6+6H_2O+2S$

(4). $As_2Cl_6+3H_2S=As_2S_3+6HCl$

The "Minimetric" Estimation of Carbonic Acid.—G. Lunge and A. Zeckendorf, Ztschr. angew. Chem. 1889, 1.

The authors wish to change the statement made in Ztschr. 1888, p. 395, that phenol-phtalein might be used either in the powder or as an alcoholic solution, as they have found that the solution undergoes a change, giving results that are too high, and recommending the phenol-phtalein to be used as a powder and dissolved in the concentrated soda solution, by washing, and also to call attention to the fact that the distilled water used in making the soda solution must first be freed from carbonic acid.

J. E. W.

Determination of Lithium in Mineral Water.—A. Carnot, Bull. Soc. Chim [3] **1**, 283.

Lithium fluoride is much less soluble in water than the fluorides of sodium, potassium, caesium and rubidium. It is still less soluble in water containing ammonia and ammonium fluoride, and by taking advantage of this property, lithium may be separated from the alkalies and determined.

*See also this Journal, **2**, 189.

†Ann. Chem. phys., 3 Serie, **13**, 44.

The mineral water is first evaporated and the alkaline earths, silica, iron, magnesium and sulphuric acid separated, and finally the ammoniacal salts driven off. Each precipitate formed is examined spectroscopically for lithium before rejecting it. The filtrate containing the alkalies, with perhaps a little magnesium chloride, is then evaporated almost to dryness, stirring constantly to prevent the formation of large crystals, strong alcohol then added, and, after grinding with a pestle and allowing to stand some time, the solution is filtered off. The saline residue is tested for lithium. The alcohol is distilled off from the filtrate and the residue dissolved in a little water to which a few drops of hydrochloric acid have been added, again evaporated and treated, still moist, with concentrated alcohol. The residue is washed with alcohol containing ether. The alcohol is distilled off and the residue consists of lithium chloride with small amounts of the other alkaline chlorides. This is dissolved in water, poured into a weighed porcelain capsule and evaporated to a few cubic centimeters. A solution of ammonium fluoride, and strong ammonium hydroxide are then added until the bulk of solution reaches 15 to 20 cc., according to the amount of lithium present. The mixture is allowed to stand over night; during the night the lithium fluoride separates and subsides. In the morning the clear liquid is poured on a very small filter and the precipitate washed several times by decantation with a mixture of ammonium fluoride and hydroxide. Care must be taken to get as little of the precipitate as possible on the filter. The filter is then dried and ignited, the capsule gently ignited and the filter ash, together with the lithium fluoride remaining in the capsule, treated with sulphuric acid, ignited to drive off the excess and the lithium sulphate weighed. The filtrate and washings, the total volume of which should not exceed 30–50 cc., contain a small amount of lithium, averaging 2 milligrams of lithium chloride, 4 milligrams sulphate or 1 milligram lithium for every 7 cc., for which an allowance must be made.

The ammonium fluoride found in commerce is very impure and must be purified before using. This is done by adding ammonium hydroxide to the solution, boiling and filtering from the silica which separates.

Detection of Adulteration in Minium.—R. Frühling, Ztschr. angew. Chem. 1889, 67.

The sample is treated with dilute nitric acid, which dissolves the monoxide of lead and leaves the peroxide as a brown powder. On the addition of sugar, alcohol or oxalic acid this is reduced to the monoxide and is then dissolved.

Foreign substances, such as heavy-spar, alumina, brick-dust, etc., are left undissolved, and may be filtered off and weighed. Should carbonate of lead or of barium be present, these will show an effervescence when the dilute acid is added. J. E. W.

Determination of the Basicity of Acids.—F. Fuchs, Monatshefte f. Chem. **9**, 1132. When acids, even very weak ones, are brought in contact with sodium or potassium sulphydrate, sulphuretted hydrogen is expelled, the amount driven off being determined by the basicity of the acid and the amount used. The author finds that the alkaline sulphydrates lose sulphuretted hydrogen in a vacuum, and it is therefore necessary to make the determination in an atmosphere of sulphuretted hydrogen and under such conditions that the temperature and pressure shall not vary during the course of the experiment. The apparatus used is a very small Erlenmeyer flask, the smaller the better, containing a ten per cent solution of caustic soda saturated with sulphuretted hydrogen. The cork of the flask has two openings, one for an almost capillary delivery tube and the other large enough to admit a small test tube. The tube to contain the substance is made of a small, short test tube and contains about 0.05 gms. of the acid. This tube is thrust into the large opening of the cork from below, open end up, just far enough to hold it. Into the same hole, from above, a glass rod is thrust until the two touch. About one-fourth gram of oxalic or tartaric acid is now thrown into the flask, which is then quickly closed. The acid drives off some sulphuretted hydrogen which fills the flask. The end of the delivery tube is then dipped in a small beaker containing sodium hydroxide solution, and the rod is forced down; this drops the acid into the sodium sulphydrate and sulphuretted hydrogen is given off and collected in the sodium hydroxide. The sulphuretted hydrogen collected in the beaker is measured by diluting to half a liter, neutralizing with acetic acid and titrating with iodine

and starch. $2I = 2H_2S = 1H$. A slight error is caused by thrusting down the glass rod which may be determined by a blank experiment and allowed for. The sulphuretted hydrogen given off may also be measured, but this can only be done indirectly since the gas is soluble in water and acts on mercury. For these reasons the principle applied in Victor Meyers' vapor density determination is used. The delivery tube is expanded into a wide tube narrowed at both ends to a capillary. Dry air is drawn through the delivery tube which is then inserted in the cork, the acid introduced and the air driven over (which is equal in volume to the sulphuretted hydrogen liberated) collected over water and measured in the ordinary way. If the tube containing the acid is of small bore the solution will not run into it readily. This difficulty may be avoided by making a hole in the wall, half way down.

Volumetric Estimation of Nickel.—Thomas Moore, Chem. News **59**, 160. This process depends on the formation of the double cyanide of nickel and potassium in alkaline solutions on the addition of potassium cyanide. Cupric ferrocyanide is used as an indicator. No change in color in the copper compound takes place until all the nickel is converted into the double cyanide, when a very small quantity of potassium cyanide immediately destroys the copper salt, showing the end of the reaction. The solutions used are (1) pure potassium cyanide, 22.5 gms. per liter, standardized with nickel solution of known strength (20 cc. = about 0.1 gm. nickel), (2) cupric ferrocyanide, made by dissolving 2.25 gms. cupric sulphate in one liter of water and adding solution of potassium ferrocyanide until it ceases to produce a further precipitate. Thoroughly shake before using. In using the process, if the nickel solution contains much free ammonia, hydrochloric acid is added to neutralize part of it; if the solution is acid, ammonia is added till strongly alkaline. Run in the potassium cyanide from a burette until a yellow tint appears in the liquid, then add a measured quantity of cupric ferrocyanide until it imparts a distinct color to the solution, and continue adding the cyanide until one drop causes a distinct change of color. A list of comparative results is appended.

Electrolytically.			*Volumetrically.*		
6.55	per cent.	Ni.	6.49	per cent	Ni.
8.60	"	"	8.70	"	"
6.35	"	"	6.37	"	"
9.55	"	"	9.60	"	"
7.85	"	"	7.77	"	"

A. H. W.

The Analysis of Subtances Containing Alumina, Lime, and Magnesia.—L. Blum, Chem. News **59**, 128 (from Ztschr. anal. Chem.) The author defends his assertion, that, in the separation of alumina, ferric oxide and phosphoric acid and lime and magnesia by precipitation with ammonia, a slight excess of ammonia is preferable to a prolonged boiling to expel it, since in the latter case the ammonium chloride splits up into ammonia and hydrochloric acid. A. H. W.

On the Separation of Zinc and Cobalt.—H. Baubigny, Chem. News, **59**, 149. (Comptes Rendus **108**, 450).

The separation of these two metals by sulphuretted hydrogen in an acid solution is accurate only when there is little cobalt in proportion to the zinc, 5 to 6 per cent. at the most. Otherwise some of the cobalt is carried down with the zinc and the precipitate takes a watery green color from the presence of a compound of the two sulphides of zinc and cobalt. Better results are obtained if there is present at the begining of the treatment a decided amount of free sulphuric acid, cobalt and zinc, even as sulphates, cannot be accurately separated by hydrogen sulphide in the presence of a small excess of sulphuric acid, unless the cobalt present be relatively small, or unless there be little zinc. The author promises to give later the details of a method of separation of zinc from nickel and cobalt in the dry way, which depends upon the absolute fixity of nickel and cobalt chlorides at the boiling point of sulphur, while zinc chloride has a considerable vapor-tension at the same temperature. A. H. W.

Estimation of Insoluble Phosphates.—Vincent Edwards, Chem. News, **59**, 159. 5 gms. are taken and treated in the usual way the water solution being tested for soluble phosphates. The residue, filter and all, is then dissolved in dilute HCl and HNO_3 in a beaker on a water bath. After treatment for half an hour the

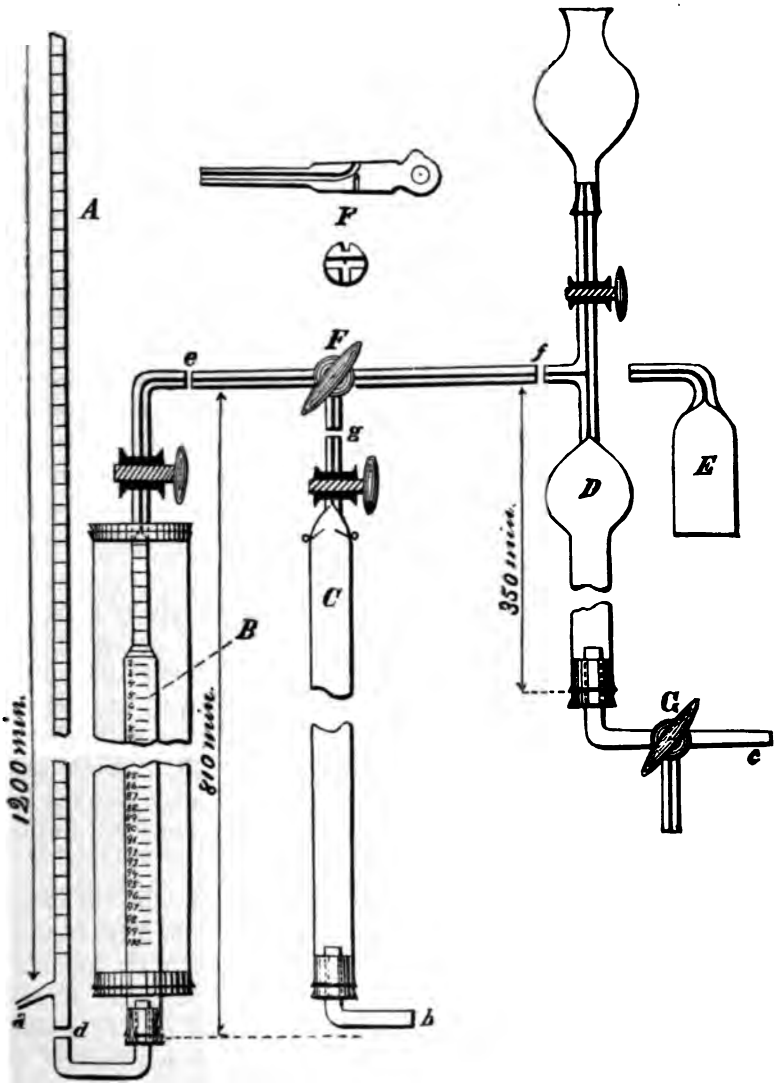
solution is filtered, 20 cc. of a solution of calcium phosphate in dilute nitric acid is added (5 gms. $Ca_3P_2O_8$ to 1 liter) and the whole diluted to 250 cc. 50 cc. of this solution (= 1 gm.) is now taken, ammonia added till alkaline, then it is made acid with acetic acid and titrated with uranium solution, heating to boiling before the first addition of uranium. The quantity of uranium required to precipitate one-fifth of the calcium phosphate solution is deducted from the result. An allowance is made for iron and alumina, and 0.5 per cent. is generally added by the author for phosphate in combination with these bases. A. H. W.

Detection of Nitrous Acid in Potable Water.—F. Musset. (Chem. Zeit. Rep. **13**, 102) finds that bacteria will give the blue color by liberating iodine when the iodide of zinc, starch solution, and acetic acid test is used for nitrites in water. To remedy this he distills the water, after decomposing the nitrites, and tests for nitrous acid in the distillate. S. C.

GAS ANALYSIS.

Improved Form of Gas Apparatus for Water Analysis.—J. J. Willard (Am. Chem. J. **10**, 53).

"*A* is a pressure tube graduated in millimeters. *B* is the measuring tube holding about 120 cc., 100 cc. of which is graduated to tenths, beginning with the stopcock *F*. Its upper part is narrow, thus admitting accurate measurement of small amounts of gas as well as large. It is enclosed in a water jacket which must be provided with some means of securing a uniform temperature throughout. *C* is the explosion tube and is ungraduated. *D* is the absorption tube surmounted by a funnel for the introduction of reagents. *E* is a laboratory vessel of the ordinary kind which may be attached instead of *D* if desired. It is obvious that any form of absorption pipette may be attached at *C*. *B*, *C* and *D* are connected by the stopcock *F*, a four-way cock shown in section above. By means of this cock, the others being suitably arranged, either tube may be put in connection with either of the others, or with the external air, without disturbing the other tube or tubes. *G* is an ordinary three-way cock. Reservoirs not shown in the cut are attached to the tubes by means of rubber



tubing at *a*, *b* and *c*. The ends of tubes are closed by rubber stoppers, and the several parts are connected by heavy rubber connectors at *d*, *e*, *f*, and *g*. The ends of the capillary tubes are ground squarely at these joints so that they come together perfectly.

The apparatus is firmly attached to a suitable support, such as any one with a little ingenuity may devise. It is essential that *A* and *B* be so supported that their relative position shall remain unaltered. The apparatus was designed for use with mercury, but water may be used. With water the pressure tube would be unnecessary.

"Concerning the mode of operating the apparatus but little need be said. It is most convenient to use Frankland's method of measuring, in which the gas operated upon is always brought to the same volume, or to an aliquot part of the original volume, by adjusting the pressure of the mercury in *A*. Points on the pressure tube corresponding to a number of convenient volumes in the measuring tube must be previously determined with care. The tensions exerted by varying amounts of gas brought to the same volume will be proportional to the amounts of gas present. If the gas is brought to an aliquot part of the original volume, the tension found may be reduced to that corresponding to the original volume by a very simple calculation.

"The explosion of the gases is performed under reduced pressure according to the principles developed by Thomas.* This method of explosion is very advantageous and may be used with this apparatus, even if water is used in the tubes *B* and *D*, by filling *C* with mercury. On lowering the reservoir connected with *C* any desired degree of rarefaction may be produced. It is convenient to have a rough scale back of *C* and extending below, for use in measuring the pressure under which the gas is confined during explosion.

"The fourth way in the stopcock *F* is essential for the discharge of the reagents employed in *D*; it is also used for the introduction of the gases. If it is desired to preserve a portion of the gas in *D* while another portion is being measured and exploded in *B* and *C*, it is necessary to close this external opening of *F*. This may be done simply and perfectly by filling the way with water or mercury from one of the tubes, and then slipping a short place of rubber tubing filled with water over the end of the stopcock and closing it with a clamp."

* Jour. Chem. Society **35**, 213.

Incompleteness of Combustion in Gaseous Explosions.—Prof. Harold B. Dixon and H. W. Smith, Chem. News, **59**, 65.

Experiments were made to determine the conditions which affect the amount of the explosive residue after gaseous explosions. The first experiments were made in a leaden tube 100 m. long and 9 mm. in diameter, having an internal surface of 29,000 cm. CO_2 was admitted at one end of the tube after each explosion until the pressure was equal to that of the atmosphere, and then one liter was displaced and collected over caustic soda solution, and the first liter was found to contain practically all the unburnt residue, which was then analyzed. Tubes of different capacities were used to ascertain the influence of the amount of surface upon the result, but it was found that the amount of surface exposed has no great influence, and hence the incompleteness of combustion cannot be attributed to the cooling action of the surface of the vessel. A. H. W.

A Convenient Form of Gas Receiver for use in water analysis has been described by F. A. Wilber (Am. Chem. J. **9**, 418). This is a modification of Cooper's receiver. The long tube has a stop-cock with large opening inserted about one-third of the distance from the upper end, which is left open. The gas is collected over mercury and measured by attaching a side tube to the lower leg into which mercury is poured until the level is the same as in the measuring tube. The absorbing solutions are introduced through the stop-cock above the mercury.

Titration of Small Amounts of Gases in Mixtures.—Behrend and Kast, Dingler's poly. J. **270**, 423.

1. Sulphuretted Hydrogen.—The gas is treated in a Bunte's burette with successive small portions of a solution of iodine until the solution is no longer discolored. If great accuracy is desired a few drops of starch solution may be introduced in the burette at first and then iodine solution run in until the starch remains blue. The iodine solution contained 1.134 gms. iodine per liter, of which one cubic centimeter is equal to one-tenth centimeter sulphuretted hydrogen gas at 0° and 760^{mm} pressure. The method is especially useful in the examination of illuminating gas and gives good results.

2. *Ozone.*—Potassium iodide solution is first run into the burette, then dilute sulphuric acid and the burette shaken. The ozone liberates a corresponding amount of iodine and this colors the solution brownish-red. A titrated solution of sodium thiosulphate (about $\frac{n}{20}$ to $\frac{n}{50}$) is then run in until the color is discharged and the amount used noted. The ozonizer and the burette are connected with an inverted U tube and rubber corks. The corks are covered with mercury, which acts as a seal and shields the corks. As an additional precaution, the tubes are thrust well through the corks. [Whether this will sufficiently protect the ozone from alteration may be doubted, since mercury also is attacked. E. H.]

IRON AND STEEL.

The Use of Hydrogen Peroxide for the Detection of Metals of the Iron Group.—Adolph Carnot, Chem. News. **59,** 15 and Comptes Rendus **107,** 997. (1.) Chromium—Hydrogen peroxide, in a hot alkaline or ammoniacal solution oxidizes chromic oxide to chromic acid. A dilute solution, say of the chloride, is heated to 100°, a few cc. of the reagent are added, and then ammonia in excess, and the whole is boiled until ammonium chromate is formed. The chromium may then be determined volumetrically either with hydrogen peroxide, or some other reducing agent, but first the peroxide remaining in the solution must be dispelled by boiling for a few minutes. Chromium may also be determined by adding to the slightly acid solution, after reduction with hydrogen peroxide, first phosphate, then sodium or ammonium acetate, and boiling for ten or fifteen minutes. In the acid solution no oxidation takes place and there is a green precipitate of hydrated chromium phosphate.

(2.) Manganese—Solutions of manganous salts are peroxidized by ammonia and hydrogen peroxide, and after complete separation of the bases in the liquid from the precipitate by repeated precipitations, it is determined volumetrically.

(3.) Iron—This metal is readily peroxidized by hydrogen peroxide in an acid solution and is thus determined. A. H. W.

The Use of Hydrogen Peroxide for the Determination of Metals of the Iron Group.—Norman McCulloch, Chem. News, **59**, 35.

Objection is made to M. Carnot's method for manganese, that the manganic hydroxide obtained, approximates in composition to Mn_7O_{13}, and is not fixed in composition, allowing a possible variation in volumetric determinations of six per cent.

A. H. W.

Reduction of Bromide of Iron Solution by Boiling.—L. L. de Koninck, Ztschr. angew. Chem. 1889, 149.

In making titrations with stannous chloride in solutions of bromide and of chloride of iron containing the same amount of iron, the author found that the bromide took about 10% less of the tin solution than the chloride. A test with ferricyanide of potash showed the presence of some ferrous salt in the bromide solution. This the author attributes to the reduction of the bromide of iron by boiling, during solution of the iron in the hydrobromic acid, as he finds that a solution of bromide of iron when boiled decomposes according to the equation $Fe_2Br_6 = Fe_2Br_4 + 2Br$, therefore on preparing a bromide of iron solution the only safe plan to follow is to dissolve the iron in presence of an excess of bromine, then expelling this by passing a strong stream of air or carbonic acid through the liquid until the escaping gas fails to color iodide of starch paper blue. This will take considerable time, as the solution of iron holds the last trace of bromine very persistently.

J. E. W.

Titration of Iron in Hydrochloric Acid Solution.*—C. Reinhardt (Chem. Zeit. **13**, 324) obtains a sharper end reaction by using free phosphoric and sulphuric acids together with the manganese sulphate. The solutions are made as follows :

1. *Potassium permanganate* containing 6 gms. $KMnO_4$ to 1 liter of water.

2. *Phosphoric acid solution.*—200 gms. of crystallized $MnSO_4$ are dissolved in 1 liter of water containing a few cubic centimeters of dilute sulphuric acid, and filtered. 1 liter of phosphoric acid (sp. gr. 1.3) is mixed with 600 cc. of water and 400 cc. of sul-

*See this Journal, Vol. **1**, 248.

phuric acid (sp. gr. 1.8) when cold this is added to the manganese sulphate solution.

3. *Mercuric chloride solution* containing 50 gms. $HgCl_2$ to 1 liter.

4. *Stannous chloride solution.*—120 gms. of granulated tin, free from iron, are dissolved in 500 cc. hydrochloric acid (sp. gr. 1.19) and after diluting to 1 liter the solution is filtered through purified asbestos. To the filtrate are added 1 liter of hydrochloric acid (sp. gr. 1.124) and 2 liters of water. ½ to 1 gm. of ore is dissolved in 25 or 30 cc. of concentrated hydrochloric acid and carefully reduced, by the stannous chloride solution. 60 cc. of the mercuric chloride solution are added, and also 1½ liters of water containing 60 cc. of the phosphoric acid solution. This last solution must be slightly reddened with the permanganate before adding to the iron solution, since it has some reducing action. The titration is then performed in the usual manner.

S. C.

Action of Finely Divided Metals on Solutions of Ferric Salts.—Carnegie (J. Chem. Soc., **106,** 464) uses the following method for the reduction of ferric solutions: Fine zinc dust is placed on the bottom of a beaker and a nearly neutral solution of ferric iron added and vigorously shaken. After the addition of a known volume of dilute sulphuric acid the agitation is repeated. To separate a certain volume of liquid from particles of zinc reverse filtration is employed. The iron is determined by decinormal permanganate solution and the amount calculated on the original solution. The action is shown by the equation

$$Fe_2Cl_6 + Zn = ZnCl_2 + Fe_2Cl_4.$$

The action is instantaneous, an estimation being made by means of zinc dust in three or four minutes, which would take an hour or more as generally performed.

When iron, mercury, silver, aluminum and copper in the finely divided state were used in place of the zinc, the rapidity of the reduction varied greatly. C. W. M.

Determination of Silicon in Ferro-Silicon.—H. J. Williams (a paper read before the A. I. M. E., New York Meeting, Feb., 1889). Pig irons, containing about 7% silicon, are insol-

uble in acids, with the exception of sulphuric acid of sp. gr. 1.25 in which they dissolve very slowly on heating. The solution of these alloys may be effected by fusing 1 gm. with sodium carbonate for 15 or 20 minutes over the blast-lamp. The fused mass is then treated with water and excess of hydrochloric acid, evaporated to dryness, and the silica filtered off and washed in the usual way; so obtained, it is perfectly white and pure. The author gives a number of analyses in duplicate and triplicate by this method which correspond closely. E. H.

Committee* Appointed to Consider the Best Method of Establishing International Standards for the Analysis of Iron and Steel.

Objects.

It is proposed that the Committee shall coöperate with other similar committees in the more important iron-producing countries, in order to provide standard specimens of iron and steel, the chemical composition of which shall have been carefully determined. The specimens adopted as standards shall be intrusted to some recognized official authority, such as the Standards Department of the Board of Trade, and shall be used either for reference in the determination of the accuracy of any proposed method of analysis, or for controlling the results of analyses in any cases of importance which may from time to time arise.

Suggestions.

1. Professor J. W. Langley, of the University of Michigan, U. S. A., to be requested to superintend the production of the samples; that they be prepared and preserved in accordance with the directions to be furnished by the Committee, and that an equal portion of each sample be forwarded to the several Secretaries of the respective Committees in the United Kingdom, America, France, Germany, and Sweden.

2. The specimens which are eventually to be adopted as standards to be supplied to not more than seven representative chemists of repute in each of the countries above mentioned, who

*British Association for the Advancement of Science, Section B. Committee:—Prof. Roberts-Austen, F. R. S. (Chairman), Sir F. Abel, C. B., F. R. S., Prof. Langley, Edward Riley, G. J. Snelus, F. R. S., John Spiller, Prof. Tilden, F. R. S., and Thomas Turner (Secretary).

shall be requested to analyze the specimens by any method or methods they may prefer.

3. In the event of the analyses giving results, which in the opinion of the Committee may be regarded as sufficiently concordant, the means of the analytical results of each of the several constituents to be adopted as representing the composition of the standards. The report on the analytical results not to be issued before the various analysts, to whom the samples have been submitted, shall have had an opportunity of examining it. The standards shall hereafter be distinguished only by letters or numbers.

4. The attention of the Committee to be for the present confined to four samples of steel, selected as containing as nearly as possible 1.3, 0.8, 0.4, and 0.15 per cent. of total carbon respectively. In addition to the determination of the amount of carbon present in each condition, the phosphorus, sulphur, silicon, manganese, and chromium also to be determined.

5. 150 kilos. of the samples selected for examination as standards to be prepared in all. This would give, after allowing sufficient for the required analyses, quite 5 kilos. of each standard for each of the five countries interested, allowing say 10 gms. for each applicant who may desire to use the standards, this would permit of 500 appeals to each of the four standards in each country, or at least 10,000 appeals in all.

6. The samples to be analyzed in the United Kingdom by W. Jenkins, Dowlais; Edward Riley, London; J. E. Stead, Middlesbrough; The Royal School of Mines; G. S. Packer, of the Steel Company of Scotland, and two others.

The metal of which the samples are to be produced to be cast in small ingots, special care being taken to prevent any irregularity of composition. After the removal of the outer skin the metal to be cut by a blunt tool in the form of thin shavings, then crushed, sieved, and intimately mixed.

8. The samples thus prepared to be preserved in separate small quantities (say of 30 gms. each), which shall be hermetically sealed in glass tubes so as to prevent oxidation.

9. These preliminary arrangements of the Committee to be communicated to the leading technical journals.

MINERAL ANALYSIS.

EDITED BY JOHN EYERMAN.

Mangano-Magnesian Magnetite.—A. H. Chester (Min. Mag. March, 1889) describes this new variety from New Zealand, where it occurs associated with serpentine. The following analysis is by F. T. Cairns.

Fe_2O_3	69.70	—	.436	.451
Mn_2O_3	3.32	—	.015	
FeO	20.50	—	.285	.431
MnO	1.50	—	.021	
MgO	4.98	—	.125	

Color; iron-black Sp. gr. 4.67

"From these figures the mineral is seen to be a magnetite in which part of the Fe_2O_3 is replaced by Mn_2O_3 and part of the FeO [illegible]O and MgO."

[illegible]rtierite fr[illegible], Arizona.—An analysis of this [illegible] given by [illegible]ler and Whitfield (Am. J. Sci. **37**, [illegible]

Al_2O_3	[illegible]	[illegible]O_3	P_2O_5	H_2O	
4.49	[illegible]	[illegible]5	0.20	1.72	99.35

[illegible]te and [illegible]m Pennsylvania.—J. Eyer-[illegible] N. Y. [illegible]89, p. 57) describes the oc-[illegible] analy[illegible]wo minerals from the French [illegible] Pete[illegible] Pa.

Stilbite.

SiO_2	58.00
Al_2O_3	13.40
Fe_2O_3	trace
CaO	7.80
MgO	1.40
K_2O	1.03
Na_2O	trace
H_2O	18.30
	99.93

Riebekite, a New Hornblende Mineral.—Sauer (Zeit. d. deuts. g. Gesellsch, 40, 138) has analyzed this new variety of hornblende from Socotra.

SiO_2	Fe_2O_3	FeO	MnO	MgO	CaO	N_2O	Ka_2O	
50.01	28.30	9.87	0.63	0.34	1.32	8.79	0.72	99.98

Elaterite, a Mineral Pitch from Derbyshire, Eng.—I. Macadam (Min. Mag. March, 1889) gives an analysis of this new mineral pitch.

C	H	O	N	S	
83.624	11.186	4.781	0.172	0.237	100.00

Sperrylite, a New Mineral.—H. L. Welles (Am. J. Sci. 37, p. 67) gives a description and analyses of this new and very interesting mineral, a platinum arsenide. It was found at the Vermillion mine, Algoma, Ontario.

Color; tin white. Crystallization; isometric; pyritohedral (S. L. Penfield).

The following is the mean of two analyses:

As	40.98	—	.546	.550=2
Sb	0.50	—	.004	
Pb	52.57	—	.267	.274=1
Rh	0.72	—	.007	
Pd	trace			
Fe	0.07			
SnO_2	4.62			
	99.46			

Beryllonite, a New Mineral.—Dana and Wells (Am. J. Sci. 37, p. 23) describe this new mineral from near Stoneham, Me. Luster; vitreous and brilliant. Colorless to white. H. 5.5–6 sp. gr. 2.845. Mean of six analyses.

P_2O_5	55.86	—	.392	=	1
BeO	19.84	—	.787	=	2
Na_2O	23.64	—	.381	=	1
Ign	0.08	—			
	99.42				

Formula Na_2O. 2BeO. P_2O_5 or $NaBePO_4$

ORGANIC ANALYSIS.

Determination of Free Alkali and Free Fatty Acid in Soap.—(Chem. Zeit. Rep. **13**, 102.) To a solution of 1 part of soap in 20–50 parts of water, sodium chloride is added until a little remains undissolved. The soap is then filtered off and washed with a saturated solution of sodium chloride, after which it is redissolved in water and the above operation repeated. The united filtrates are titrated for free alkali with $\frac{n}{10}$ sulphuric acid, using phenol-phthalein for an indicator.

The soap, after the second washing with the sodium chloride solution, is dissolved in 30 cc. of absolute alcohol by heating on a water bath and then titrated for free acid with $\frac{n}{100}$ caustic potash solution, using a few drops of the same indicator. Since the alcohol has a slight action on the phenol-phthalein it is best to titrate 30 cc. of the alcohol containing a few drops of the indicator with the potash solution and deduct it from the analysis. S. C.

Detection of Sugar in Logwood Extract.—O. Schweisinger, (Chem. Zeit. Rep. **13**, 41).

Logwood extract is frequently adulterated with sugar, and to detect this 3 to 5 gms. of the extract are dissolved in 50 cc. water and this solution is thoroughly shaken with 10 cc. of basic acetate of lead solution. After standing a short time, it is filtered and the filtrate put in a 100 mm. tube and polarized. The remaining filtrate is heated with HCl to precipitate the lead and heated with an inverted condenser for half an hour and then allowed to cool. The slight excess of acid is neutralized with Na_2CO_3 and the solution filtered and titrated with Fehling's solution. S. C.

Some Investigations on Milk.—J. N. Zeitler, Ztschr. angew. Chem. 1889, 13.

The author gives results of analyses, made at the Connstatt experimental station, of milk given by cows during the time they were restricted to certain kinds of food and doing draught service.

J. E. W.

The Chemistry of the Flax Fiber.—C. F. Cross and E. J. Bevan. Chem. News **59**, 135. The pure fiber substance is a compound of cellulose with a pectosic group as determined by Kolb.

The action of HNO_3 (1.15 sp. gr.) forms a quantity of mucic acid evidently from the non-cellulose constituent which amounts to from 5 to 10 per cent. Oxalic, and carbonic, fatty and waxy acids are also produced by the use of HNO_3. After separation of the latter acids by exhaustion with alcohol, an oxycellulose is obtained, comparatively pure. Potassium permanganate in the presence of an excess of caustic soda produced from pure bleached linen (flax cellulose) a similar oxycellulose (45 per cent), 18 per cent of oxalic acid and 15 per cent. of a soluble carbohydrate which yielded furfurol on hydrolysis with acids. Hot alcohol dissolved out from the scutched fiber 2 to 3 per cent. of a mixture composed of, (a). a wax, a mixture of a free wax alcohol ($C_{23}H_{47}OH$) with a ketone-resin compound of the same alcohol, and, (b). in the filtrate from the wax, a variety of constituents, viz. (1). a soluble carbohydrate of the ligno-cellulose type and (2). a green semi-fused residue which after the extraction of the carbohydrate is resolved by benzene into two constituents a brown residue or wax (resembling that under (a)) and a bright green oil which becomes semi-solid on removal of the solvent. A. H. W.

Some Reactions of Vanadyl Sulphate upon Glucosides and Alkaloids.—Fr. Kundrat, (Chem. Zeit. **13**, 265).

The author recommends as being more decided in action than Frohde's well known reagent, a solution prepared by Johnson* by dissolving ammonium vanadate in concentrated sulphuric acid so that 10 cc. of acid will contain 0.1 gm. vanadyl sulphate.

The results he obtained are as follows:

Substance.	*Color with Vanadyl sulphate solution is:*
Aconitine	Light Coffee Brown.
Atropine	Red, but gradually changes to the original yellow solution.
Apomorphine	Dark violet blue, which changes immediately to a dirty green and afterward to a reddish brown, and finally to a light brown. Gas is evolved when the substance is dissolved.
Brucine	An intense blood red, which gradually disappears.
Cinchonine	Slightly orange, which assumes a greenish tint after standing a long time.
Cocaine	Orange. Foams during solution.
Codeine	Greenish brown, which passes gradually into a brown.
Colchicine	Green, changing gradually into a coffee brown.
Conine	An intense green, which after a time assumes a brownish tint.
Digitaline	An intense dark brown with a reddish cast, which disappears after a time.
Quinidine	A slight bluish green.
Quinine	Slightly orange, passing to a bluish green and finally to a greenish brown.

*Ztschr. anal. Chem. **24**, 154.

Caffeine	No reaction.
Morphine	Brown, similar to the digitaline reaction.
Narceine	Brown, changing to a dirty blue-violet with a brownish cast, and finally to a reddish brown.
Narcotine	An intense blood red with a carmine cast.
Nicotine	Only slightly darker and soon returns to the original shade.
Papaverine	Violet with a brownish cast, similar to the veratrine reaction. This changes gradually to a bluish green and finally to an orange yellow.
Picrotoxin	Only slightly darker. Fragments of picrotoxin give the solution a little stronger yellowish red color.
Pilocarpin	Light orange.
Piperine	From an intense reddish brown to a very dark brown. Fragments moistened with the reagent become black.
Physostigmine	Greenish yellow, but soon changes to carmine and finally to a yellowish brown.
Salicylic acid	Brownish green, which becomes darker on standing,
Antifebrine	Carmine, which immediately becomes brown.
Antipyrine	An intense greenish blue, which soon loses the greenish tinge. The reaction is similar to the one with quinine.
Kairine	A dirty rose red, changing to a light brown and then to a brownish green. The substance foams during solution.
Santonine	No reaction.
Solanine	Coffee brown. After some time the outside of a drop of the solution appears carmine, which passes through yellow to a dirty green in the centre. After two hours the drop gelatinizes and changes to a dark violet mass.
Strychnine	Bluish violet, changing through reddish violet to carmine and finally to a brilliant red.
Veratrine	Brownish red, changing gradually to a dark reddish violet.

S. C.

Some Tests for Ethereal Oils.—Anton Ihl (Chem. Zeit. **13**, 264) suggested by the action of phenol on a carbo-hydrate in the presence of an acid the author tried using the ethereal oils in place of phenol, but oil of peppermint was the only one that gave a satisfactory reaction. This when mixed with alcohol and cane sugar and heated with dilute hydrochloric acid gave a bluish green color. On further investigation he found that phloroglucine was a very sensitive reagent for the following oils which contain eugenol.

Oil of Cloves when treated with an alcoholic solution of phloroglucine and concentrated hydrochloric acid gives a clear bright red color, with an alcoholic solution of resorcin in place of the phloroglucine, the oil, by the same treatment, gives a reddish violet color, when heated with a solution of pyrogallol it gives a violet color.

Oil of Cassia, with phloroglucine gives a dark red color; with resorcin, a vermillion.

Oil of Pimento, with phloroglucine gives an intense rose red color; with resorcin, a dirty violet when heated.

These three oils give a yellow color with an alcoholic solution of aniline sulphate and hydrochloric acid; with alpha naphtylamine they give an orange yellow. S. C.

Analyses by Polarization.*—R. Pribram, Monatshefte f. Chem. **9**, 485. When acetone is added to a tartaric acid solution the rotatory power is decreased, methyl, ethyl, propyl, isopropyl and isobutyl alcohol decrease the rotatory power as well as trimethylcarbinol, octyl alcohol and allyl alcohol. Many esters behave in the same way. Tannic, acetic, propionic and butyric acids also have the same effect.

The author has also examined the behavior of benzene, mononitro-benzene, toluene, mono-nitro-toluene, mono-chlor-benzene, ethyl bromide, nitro-ethane, urea, aniline and pyridine, (Ber. **22**, 6) using ethyl alcohol as the solvent.

New Method for Organic Analysis.—W. L. Dudley, (Ber. **21**, 3172; Am. Chem. J. **10**, 433). Instead of copper oxide, manganese oxide (Mn_3O_4) is used, prepared by dissolving manganous carbonate in moderately dilute nitric acid, evaporating to dryness and igniting over a blast lamp. This is more porous than copper oxide, lighter and quite as effective. A platinum combustion tube, Fig. 1, such as is used in carbon determination in pig iron, is preferable to a glass tube. The tube shown is 48.5 cm. long and 1.5 cm. in diameter. The tail *D* is 13 cm. long and 0 5 cm. in diameter. *A* is manganese oxide, several large lumps being put in first. No channel is left in the tube as the oxide should be sufficiently coarse to allow of the passage of gas. If a glass tube is used, a slight channel should be left. The substance is introduced in the boat *B*, best of porcelain, and followed by a roll of platinum which has a loop in front so that it can be easily withdrawn. At *E* a gas-tight brass coupling is inserted; this carries a tube 0.5 cm. in diameter to which is connected the apparatus furnishing either air or oxygen as desired.

"Instead of the tube being reduced to 0.5 cm. in the tail *D*, Fig. 1, the design shown in Fig. 2 is proposed to facilitate the passage of the water into the calcium chloride tube. The tube *B* is only slightly smaller than the main body and the end is closed with a stopper carrying the stem of the calcium chloride

*See also this Journal **2**, 420, 421; **3**, 72.

tube. At *A* is placed a platinum basket to retain the manganese oxide. The whole plan of the apparatus is shown in Fig. 3. Before introducing the substance, and connecting the calcium chloride tube and potash bulb, the portion of the tube containing the manganese oxide is heated to full redness and a rapid current of dry and pure air is passed through. The burners are shut off, except the last two or three, which are sufficient to keep about

Fig. I.

Fig. 2.

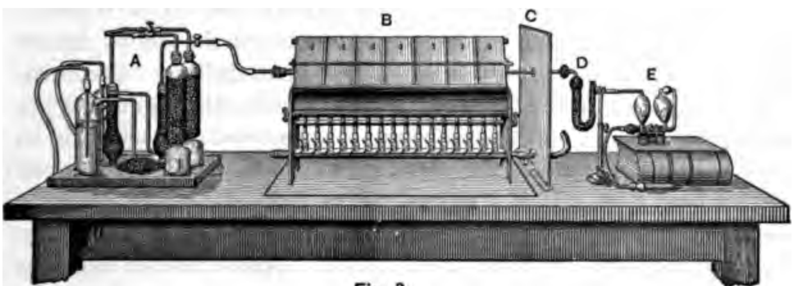


Fig. 3.

Fig. 4.

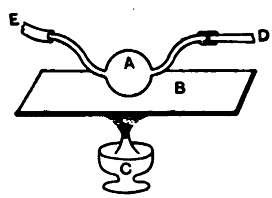


Fig. 5.

two inches of the tube red-hot. The current of air is then stopped and the calcium chloride tube and potash bulbs are attached, and finally the substance is introduced in a platinum or porcelain boat (by removing the coupling at the posterior end) followed by the roll of platinum gauze. A slow current of air is then turned on, it having been purified by passing through the apparatus shown at *A*, Fig. 3, containing, 1st, a strong solution

of caustic potash; 2d, pumice and sulphuric acid, and, 3d, solid caustic potash. Two burners under the portion of the tube containing the roll of platinum gauze are now lighted and the substance is gradually approached by lighting the burners under the manganese oxide. The red-hot platinum gauze will decompose any condensable gases, which may diffuse back if the combustion should proceed a little too rapidly, and prevent a deposit in the posterior portion of the tube outside of the furnace. An asbestos shield *C*, Fig. 3, protects the calcium chloride tube and potash bulbs from the heat of the furnace.

"I prefer to use a Geissler potash bulb with tube for solid potash attached, in which case the air or oxygen may be forced in at the rate of two or three bubbles a second. It is also desirable to use a slight exhaust as well as pressure.

"When all the burners have been lighted, the air is replaced by a current of oxygen, which is continued for about fifteen minutes, when the combustion will have been completed. The burners are then all turned off excepting the three at the end under the manganese oxide, and the bulbs are disconnected and set aside to cool. In a few minutes the platinum gauze may best be removed with a bent wire, and if a second set of bulbs be ready, another combustion may proceed at once. In this way a combustion may be made easily every hour, the actual time consumed in burning the substance being from thirty to forty minutes.

"The manganese oxide is regenerated by the oxygen which is passed in during each combustion; it is never reduced below MnO and therefore can do the platinum tube no damage. We have a platinum tube in which over fifty combustions were made during two months without removing the manganese oxide, and on examination afterward, the inside of the tube was found to be as clean and sound as ever."

Results of five combustions of sugar in the platinum tube are given with a mean result of: Carbon, 42.07 (theory 42.10); Hydrogen 6.49 (theory 6.44). In a glass tube, equally good results are obtained as is shown by another set of five combustions.

In burning liquids with high boiling point, the required amount is poured into the boat by means of the apparatus shown in Fig. 4. The bottle containing the liquid and dropper *A* are

first weighed, then the necessary amount of liquid is transferred to the boat by the dropper with rubber bulb *A*, and the whole apparatus weighed again.

For volatile liquids the bulb shown in Fig. 5 is used. A current of nitrogen (made by passing air over heated copper) is passed through the bulb and into the combustion tube, and the bulb is heated by radiation from the iron plate *B*, this arrangement prevents danger of explosion and does not interfere with complete combustion; after the liquid has evaporated air or oxygen is substituted for the nitrogen.

Combustion by Means of Chromic Anhydride.—Cross and Bevan (Journ. Chem. Soc. **53,** 889) have investigated the estimation of carbon in organic substances by oxidation with chromic anhydride and sulphuric acid. The conditions for the analysis are as follows: (1). A quantity of substance must be used yielding 90 to 100 cc. of gas; (2). 9 cc. of sulphuric acid must be employed; (3). an excess of chromic acid of about 30 per cent. beyond that required for complete oxidation is used.

The substance is dissolved in the sulphuric acid in a flask, the chromic anhydride placed in a small tube in a horizontal position in the neck of the flask, but not in contact with the acid. The flask in a horizontal position is connected with one end of a U tube. This end is graduated and is filled to the zero mark with mercury, or is adjusted to zero after the flask is connected. The tube containing the chromic anhydride is now shaken down into the acid and the whole agitated. Immediately the combustion begins, and as soon as it begins to cease the flask is heated to 100°, at which temperature the combustion is complete. The tube is allowed to cool. The temperature is taken before and after heating, and the volume of carbonic anhydride reduced to 0° and 760 mm., and from this the weight and percentage of carbon, are obtained. The results obtained were invariably too low, but when the correction for the absorption of carbonic anhydride by the sulphuric acid was added the results were quite accurate. The correction may be found by the formula

$$\frac{\text{Carbon per centage of substance}}{25} \times 0.4 = \text{correction}.$$

This is to be added to the amount found from the gas liberated.

C. W. M.

Detection of Antifebrin in Phenacetin.—M. J. Schröder (Chem. Zeit. Rep. **13**, 40). The antifebrin is first converted into aniline and then into phenol, which is tested with Plugge's reagent (a nitrous acid solution of mercurous nitrate). 0.5 gm. of phenacetin is heated in a test tube with 5–8 cc. of water and then allowed to cool. Most of the phenacetin crystalizes out and is filtered off. Some potassium nitrite and dilute nitric acid are added to the filtrate and boiled. Then a few drops of Plugge's reagent are added and the solution boiled again. The presence of antefebrin, if more than 2%, is indicated by a red color. S. C.

Estimation of Carbolic Acid.—L. de Koninck, Ztschr. angew. Chem. 1889, 119.

The author refers to an article by Williams (Jour. Soc. Chem. Ind. January, 1889) on this subject, and intimates that Williams did not rightly understand a former article published in the "Analyst" **13**, 194. Then he goes on to say that in England there are two varieties of carbolic acid—

1st. The pure preparation of the pharmacist containing 10% water, which, when shaken with a saturated solution of salt will not dissolve; also, when it is mixed with four times its volume of 10% caustic soda solution the solution will remain clear and with bromine will give a precipitate similar to chloride of silver.

2d. Commercial carbolic acid, which contains considerable cresylic acid. This when shaken with salt solution should remain clear, and if, when treated with four times the solution of 10% soda solution there remain a residue, 10 cc. of benzol is added, which collects the tar. Then, upon adding three volumes of benzol, if no water is present, the solution remains clear. J. E. W.

Quantitative Determination of Aniline and Monomethylaniline.—F. Reverdin and Ch. de la Harpe (Chem. Zeit. **13**, 407). The authors have found the two methods of analysis that are commonly used for the above determinations to be unsatisfactory, and have adopted the following:

Determination of Aniline.—7 or 8 gms. of the mixture of aniline bases to be analyzed are dissolved in 30 cc. of hydrochloric

acid diluted with 100 cc. of water. A standard solution of a sodium salt of beta-naphtol-disulphonic acid is prepared that will be equivalent to 10 gms. naphtol to 1 liter of water.

10 cc. of the solution of the aniline bases are diluted with some ice water and treated with enough sodium nitrite to convert the same amount of pure aniline into the diazo compound. The product thus formed is added, little by little, to a measured quantity of the standard solution which has been treated with an excess of sodium carbonate. The product of this reaction is precipitated with common salt and filtered off, and the filtrate is tested for an excess of diazobenzol or the standard solution. By repeating this operation the volume of the standard solution which is necessary to combine with the diazobenzol formed in 10 cc. of the solution to be analyzed, is found.

Determination of Monomethylaniline.—1 or 2 gms. of the substance is placed in flask provided with a reversed condenser and to it is added exactly twice its weight of acetic anhydride and the mixture is allowed to stand at the ordinary temperature for half an hour. 50 cc. of water are then added and heated on a water bath for three-quarters of an hour, when the excess of acetic anhydride will be completely decomposed. After cooling, the solution is made up to a known volume and the excess of acetic acid is titrated with a standard soda solution, using phenolpthalein as an indicator. The amount of monomethylaniline is calculated by subtracting from the total amount of acetic acid used the amount used by the aniline, the percentage of which has been determined by the process just described. S. C.

Determination of Sugar by Fehling's Solution.—H. Causse, Bull. Soc. Chim. **50,** 625. Potassium ferrocyanide is without action on Fehling's solution either at low or at high temperatures. If to a hot Fehling's solution ferrocyanide of potassium be added, and then a solution of sugar, drop by drop, a precipitate of cuprous oxide forms at the point of contact of the two liquids which dissolves and the solution bleaches. If the proportions of ferrocyanide and oxide are suitable we obtain, without trace of deposit, a solution entirely colorless. For the test the author uses 10 cc. of the solution, 20 cc. distilled water,

and 4 cc. of a solution of ferrocynide of potassium, 1.20, and heats to boiling in a flask. When a solution of sugar is slowly added the blue color disappears, the flask is allowed to cool, when colorless crystals separate out, which the author intends to investigate.

F. R. B.

Valuation of Rice for Starch Making.—H. Schreib (Ztschr. angew. Chem. 1888, 694). The broken rice, consisting of the broken and smaller sized grains, is used for rice making. This is examined for protein, moisture and ash; as the amount of fat, sugar, gum and cellulose varies very slightly, the sum of these constituents is added to those found for protein, ash and moisture, and the sum subtracted from 100 gives the percentage of starch. Protein or gluten is very objectionable in large amount, as it retains the starch mechanically, and for every per cent. present an equal amount of starch will be retained and pass into the offal, which is useless except for fodder or manure. For this reason, a rice with 78% of starch and 4% protein is more valuable to the manufacturer than one containing 80% starch and 8% protein.

The method given above for starch determination is preferable to the direct method for this special purpose because it is more rapid, and because the direct methods give a figure which includes the gum, sugar and soluble starch—substances valuable for fodder and to the distiller, but valueless to the starch maker.

In the process of manufacture, the rice is first treated with soda lye to swell the grains and dissolve part of the protein. The amount of soluble protein in different samples varies considerably, and since this makes a decided difference in the yield of starch obtained, it is well to know the proportion of soluble protein in the grain.

Testing the starch.—The starch is tested by mixing 4 gms. to a milky liquid with 50 cc. water and heating to a boil for 1 minute in a porcelain casserole. If the starch is of proper quality the paste will not run from the dish after it cools. In making this test only porcelain vessels should be used, since the starch is apt to burn when heated in metal, and the boiling must not be continued too long, as this causes thinning, if acid should be present.

Detection of Foreign Coloring Matter in Wine.—A. Ragnoul (Chem. Zeit. Rep. **13**, 104) claims that a soap solution will

destroy the natural color in wine without imparting the green color that other alkaline solutions do, and leave foreign coloring matter unchanged. 5 cc. of a strong soap solution in water is diluted in a test tube with an equal bulk of water and 10 to 20 drops of wine are added. If the solution, after mixing, is colorless, no foreign coloring matter is present. S. C.

Separation of Phenol and Resorcin from Salicylic Acid.—L. van Itallie (Chem. Zeit. Rep. **13**, 46). When two drops of ferric chloride solution are added to 100 cc. of a saturated solution of salicylic acid containing 2% of phenol and resorcin, a violet blue color is produced. By adding a few drops of lactic acid the violet color caused by the phenol and resorcin is immediately changed to a yellowish green, while the original color caused by the presence of salicylic acid remains unchanged. S. C.

Determination of Strychnine in the Seed Extract.—L. van Itallie (Chem. Zeit. Rep. **13**, 47). 2 gms. of the extract are slightly acidified with sulphuric acid and then dissolved in 20 cc. of water. After standing an hour, 10 gms. of lead acetate solution (1:10) are added and the solution filtered through a dry filter. 5 cc. of dilute sulphuric acid (1:25) are added to the filtrate, and after the precipitate has settled the solution is filtered and the filtrate made alkaline with ammonia. The alkaloid is then dissolved out by shaking the solution with an equal volume of chloroform and separated as a yellowish mass by distilling off the chloroform. This residue is dissolved in 15 cc. of $\frac{n}{10}$ acid and the excess of acid titrated back by $\frac{n}{100}$ soda solution. S. C.

Examination and Estimation of Pepsin.—A. Kremel (Chem. Zeit. Rep. **13**, 47). 0.1 gm. of pepsin is dissolved in 150 cc. of water and 2.5 gms. of dilute hydrochloric acid, and the solution warmed by dipping in water at 45°. 10 gms. of powdered egg albumen are added and the heat continued at the same temperature for six hours. By this time good pepsin will have the albumen completely dissolved, so that the addition of 2 cc. of conc. nitric acid produces no precipitate, and at the most only a slight turbidity. For the estimation of pepsin, 1 gm. of powdered egg albumen and 0.1 gm. of pepsin are put into a 100 cc. flask with 50 cc. of a 2% hydrochloric solution and heated at 40° for

three hours. The solution is then exactly neutralized with sodium carbonate and heated to 90° on a water bath until coagulation takes place. After cooling, water is added to the mark and the solution filtered. 50 cc. of the filtrate are evaporated to dryness in a porcelain dish on a water bath. The residue is dissolved in water and carefully filtered through a moistened paper into a platinum dish. The filter is carefully washed and the filtrate evaporated to dryness on a water bath. The residue is then dried at 100° and weighed. S. C.

Examination of Roasted Coffee for Addition of Sugar and Syrup.—Stutzer and Reitmair Ztschr. angew. Chem. 1888, 701. The coffee roasters of Bonn are in the habit of adding sugar or syrup to coffee during the roasting process. This prevents loss in weight (the gain in weight being about 5 per cent.) and adds the weight of the cheaper sugar or syrup to that of the dearer coffee. The roasters assert that this process improves the coffee and makes it go further, but the authors have proved that such addition is detrimental to every one but the seller. To detect this form of adulteration the authors proceed as follows: 20 gms. whole coffee beans are treated with 500 cc. water in a liter flask and shaken in a mechanical shaken for 5 minutes; the flask is then filled to the mark with water and immediately filtered. 50 cc. filtrate is evaporated to dryness in a platinum dish on the water bath, dried for two hours at 95° to 99°, weighed, burned, and the ash also weighed. Pure roasted coffee treated in this way gives no color to the water and yields from 0.44 to 0.72 per cent. organic extract, while syrup-roasted coffee gives a more or less strong color and from 1.81 to 8.18 per cent. organic extract. If the time of treatment is increased to 10–15 minutes the organic extract for sophisticated coffee is increased by several per cent., while for pure coffee the gain does not exceed one-half per cent. The determination of moisture is of little assistance in detecting this form of adulteration.

The authors consider the process of J. König detailed on p. 631 of the same Journal (extraction with hot water and determination of the amount of extract) less useful than their own.

The determination of extract in coffee is best made indirectly,

by weighing the residue, and for this purpose use is made of the Gooch crucible.

The Estimation of Starch.—J. N. Spence (J. Soc. Chem. Ind. **7**, 77) uses a modification of Asboth's method, which is described as follows:

"Three grams are placed in a mortar (when there is any oil or resinous bodies present they must be extracted by ether in the "Soxhlet" first), and 30 cc. of cold distilled H_2O added in quantities of 10 cc., rubbing well between each 10 cc. Then 30 cc. of boiling water are added in the same manner, and 40 cc. of boiling H_2O used to transfer the substance out of the mortar into the 250 cc. flask. The flask is then placed in a water bath and allowed to remain for half an hour, with frequent shaking (to prevent lumping). At the end of the half hour the contents will be entirely gelatinised. Immediately on taking the flask out of the bath 50 cc. of baryta water are added, the stopper replaced, and the whole well shaken for a few minutes (taking care not to let the stopper stick). I find it advisable to standardise the baryta while the starch is gelatinising, as this process will not work unless the 50 cc. of baryta water require 90 cc. or over it of decinormal HCl. Therefore if the baryta does not require so much it must be made up. After shaking, proof spirit is added up to the 250 cc. mark, and the whole well shaken (care being taken not to let the stopper fly out). After settling, 50 cc. of the clear liquid are run off and titrated in the usual way.

"Prof. Asboth recommends two drops of phenol-phthalein to be used, but this depends on the strength of the phenol-phthalein solution. I find it best to add it until a faint marked pink coloration has been produced.

"The difference between the two processes seems to lie in the fact that Professor Asboth recommends the additon of the baryta water *in the cold*, whereas by adding it *to the hot* solution a better marked compound is obtained. I find that the compound formed by the addition of baryta water to gelatinised starch is soluble in water, but is precipitated again by the alcohol; it is therefore not insoluble as stated.

"I also use the baryta never below the strength capable of saturating 90 cc. of $\frac{n}{10}$ HCl; my reason for this is, that I find it pos-

sible to form more than one compound, depending on the strength of the baryta water.

"For example, I find that starting with the baryta capable of saturating 20 cc. $\frac{n}{10}$ HCl, the compound formed contains only about 2 per cent. BaO, whereas by using baryta capable of saturating 90 cc. $\frac{n}{10}$ HCl, the compound contains about 20 per cent. BaO. Therefore by using baryta which consumes 90 cc. $\frac{n}{10}$ HCl, you supply sufficient baryta to form the proper compound, and also have a small quantity in excess. I think it is possible to form a great many compounds with baryta and gelatinised starch, all depending on the strength of the baryta. No matter how weak your baryta, you always get some left over, until you reach the strength capable of saturating 90 cc. $\frac{n}{10}$ HCl, above which there seems to be no change in the proportions of the compound."

Color Reactions of Aniline Dyes Upon Essential Oils.—John D. Riker, (Proc. Mich. State Phar. Ass. 1888, 140. "Ten or fifteen drops of the oil to be tested was placed in a small (0.5 inch diameter) test tube; then one to three drops of an aniline dye solution added and the changes noted in the cold as well as after heating.

"Below are given the results of careful observations, using oils obtained from retail drug stores in Ann Arbor, with no definite proof of their purity.

"As soon as a list of unquestionably pure oils can be obtained the writer will endeavor to verify these results.

"The dyes used were common commercial dyes obtainable at any drug store.

"All the dye solutions were made by taking 0.5 grams of the powdered dye and dissolving in 25 cc. of distilled water.

"Experiments with Magenta solution: 1–3 drops of the dye solution were added to 10 to 15 drops of the oil with the following results:

"Cassia oil takes up scarlet red color in cold; heating turns bright red; on cooling again turns scarlet.

"The oil of cloves absorbs red in cold; on heating all color disappears.

"Absorbing red in cold; no change on heating, oils of

Caraway,	Pimenta,
Croton,	Tansy,
Peppermint,	Wintergreen.

"Mustard oil absorbs red in cold; heating all color disappears, leaving the oil milk white.

"Pennyroyal takes up scarlet red in cold: on heating turns a deep ruby red.

"Magenta not absorbed with oils of

Anise,	Eucalyptus,	Lemon,	Savine,
Amber,	Fire Weed,	Orange,	Wormwood,
Bergamot,	Hemlock,	Origanum,	
Cedar Red,	Juniper Wood,	Sassafras.	

"Experiments with Berlin blue solution:

"Absorbing blue in cold; no change on heating; oils of

Cloves,	Mustard,
Caraway,	Pennyroyal,
Croton,	Tansy.

"Turning deep cherry red in cold; no change on heating; oils of

Anise,	Hemlock,
Fire Weed,	Sassafras.

"Savine oil takes up purple in cold; on heating turns ruby red.

"Cassia oil turns green color in cold; no change on heating.

"Juniper wood and Eucalyptus oils shows a play of colors. The blue solution turns purple, then colorless, while the oils takes a brown color; heating hastens the disappearance of the blue color of the solution and leaves the oil a golden yellow color.

"Wintergreen and Peppermint oils absorb blue in cold; heating slightly, turn violet; heating to boil, turn a deep cherry red; on cooling gradually, turn from cherry red to purple, then blue.

"Berlin blue not absorbed with oils of

Amber,	Lemon,
Cedar Red,	Orange,
Bergamot,	Origanum.

"Experiments with Hoffman's violet solution:

"Absorbing violet in cold; no change on heating; oils of

Clove,	Croton,	Pennyroyal,	Sassafras,
Caraway,	Lemon,	Peppermint,	Wintergreen,
	Origanum,	Savine.	

"Absorbing violet in cold; color disappears on heating; oils of

Anise,	Fire Weed,
Bergamot,	Orange,
Cedar Red,	Tansy.

"Amber oil absorbs violet in cold; heating turns deep cherry red.

"Caraway oil turns scarlet red in cold; heating no change.

"Cassia and Mustard oils turn blue in cold; heating no change.

"Pimenta oil turns dark ruby red in cold; heating no change.

"Hoffman's violet solution not absorbed with oils of Eucalyptus, Hemlock or Juniper. [Note.—Gentian violet dye gives nearly same results.]

"Experiments with Methyl green solution:

"Absorbing various shades of green in cold; heating no change; oils of

Cassia,	Mustard,
Croton,	Pennyroyal,
Caraway,	Tansy.

"Absorbing green in cold; color disappearing on heating; oils of

Anise,	Pimenta.
Fire Weed,	Cloves.

"Amber oil will not absorb methyl green in cold but on heating takes a grass green color, and the methyl green solution becomes blue.

"Peppermint oil, no absorbtion in cold, on heating takes up the green color.

"Wintergreen absorbs grass green color readily in cold and the methyl green solution turns blue; no change on heating.

"Methyl green not absorbed with oils of

Cedar Red,	Juniper Wood,	Orange,	Savine,
Bergamot,	Lemon,	Origanum,	Wormwood,
Eucalyptus,	Hemlock,	Sassafras,	Spike.

"Absorbing Nigrosin coloring in cold; no change on heating: oils of

Cloves,	Eucalptus,	Wintergreen,
Cassia,	Pimenta,	
Caraway,	Pennyroyal.	

"Not absorbing Nigrosin in cold but taking up readily on heating; oils of

Amber,	Sassafras,
Anise,	Tansy.

"Absorbing Nigrosin Comp., but slightly in cold; no change on heating: oils of Mustard, Peppermint and Orange.

"Nigrosin solution not absorbed with oils of

Bergamot,	Fire Weed,	Lemon,
Cedar Red,	Hemlock,	Origanum.
Croton,	Juniper Wood,	

"Experiments with Aniline blue solution:

"Absorbing blue in cold; no change on heating; oils of

Cloves,	Cassia,	Pennyroyal,
Caraway,		Pimenta.

"Mustard oil absorbs blue in cold, heating fades or blanches until all color disappears.

"Peppermint oil no absorbtion in cold but absorbs blue color slightly on heating.

"Sassafras oil, no absorbtion in cold; heating blanches the blue solution.

"Aniline blue not absorbed with oils of

Anise,	Eucalyptus,	Orange,
Amber,	Fire Weed,	Origanum,
Croton,	Hemlock,	Savine,
Cedar Red,	Juniper Wood,	Tansy,
Bergamot,	Lemon,	Wintergreen.

The above results would seem to indicate that further investigation in this direction might prove fruitful of reliable qualitative tests for many of the essential oils."

Estimation of Nicotine in Cigarettes.—J. H. Sharper, (Proc. Mich. State Pharm. Ass. 1888, p. 121). "The tobacco was dried, three grams of it were then weighed, macerated with water acidulated with sulphuric acid for twenty-four hours, the expressed liquid and washings concentrated, filtered, and further concentrated with washings of the filter to 50 cc. Of this, portions of 10 cc. were taken to obtain several agreeing titrations with Mayer's reagent. Of the latter, 1 cc. was taken to equal 0.00405 grams of nicotine, as nearly supported by the eight estimations of Zinoffski cited by Dragendorff. In the assay of cigarettes, 5 grams were taken for the operation, and therefore each 10 cc. represented 1 gram of the material. The results were as follows:

Brand of Cigarettes.	Mayer's Reagent for 1 gr. material.	Percentage of Nicotine.
Kinney Brothers' "Full Dress"	2.7 cc.	1.10
Kinney Brothers' "Satin"	3.1 "	1.26
"Richmond Straight Cut"	3.4 "	1.38
"Old Judge"	3.8 "	1.54
"Temple Bar"	3.7 "	1.50
"Sweet Caporal"	4.0 "	1.62
"Virginia Bright"	4.2 "	1.70
"Duke's Cameo"	4.4 "	1.78
"Michigan Leaf Tobacco"	4.8 "	1.94
"New York Leaf Tobacco"	4.2 "	1.70
"Kentucky Leaf Tobacco"	4.6 "	1.86
"Richmond Gem Smoking"	3.9 "	1.58

Some estimations of the quantity of alkaloid remaining undecomposed in the smoke of cigarettes were made by drawing cigarette smoke through acidulated water. The proportion of alkaloid of the smoke so obtained, estimated as nicotine, was in the average a little over one-half the total alkaloid found in the same cigarettes.

DISCUSSION.

PROF. PRESCOTT: Investigations have shown several times that a proportion of nicotine of the tobacco remains undecomposed when tobacco is smoked. So if tobacco smoke is drawn through acidulated water this undecomposed nicotine will be recovered

again, in quite a considerable, though a variable portion. Of that portion of nicotine which is decomposed some part is resolved into organic derivatives of nicotine, the physiological effects of which have never been determined, the other portions are decomposed into final harmless products. I was interested, however, in the statement made in a discussion upon a paper before the American Association for the Advancement of Science at Cleveland week before last. It was a statement made by Prof. Dudley, of Nashville, that he had analyzed the smoke of cigarettes regarding their proportion of carbon monoxide, and he found it large, and he said that from a good deal of study of the subject he was disposed to attribute the especial injury from cigarette smoking to the inhalation of the smoke containing monoxide of carbon. Both cigarette smoke and cigar smoke, he said, contain carbon monoxide. But cigarette smokers inhale a much larger proportion of the smoke than do smokers of cigars and pipes, an important element in the effects of cigarette smoking."

Testing Acetanilid (Antifebrin).—S. Kidder, Jr., Trans. Mich. Pharm. Ass. 1888, 158.

"The following reactions, made the subject of experiment, the writer did not find any literature upon:

"To a moderately concentrated alcoholic solution of acetanilid there were added a few drops of a neutral solution of permanganate of potassium. The permanganate was gradually reduced to the manganate, the solution taking a decided green color. By cooling the test-tube under the hydrant for a few moments, a voluminous precipitate formed and soon solidified into a gelatinous mass, solid enough to maintain itself in the test-tube when inverted. The test applied in the same way to a solution of the permanganate made strongly alkaline with potash gave nearly the same results, except that the reduction was slower and the precipitate did not solidify. But when the permanganate was made strongly acidulous with the sulphuric acid, the permanganate was at once reduced to the dyad state, and a red precipitate appeared. Stannous chloride had no effect; neither had nascent hydrogen; nor the other common reducing agents. These tests would seem to indicate acetanilid to be a reducing agent of some capacity.

"The following tests were applied precisely as if testing for an alkaloid in the dry way, that is, by touching the end of a glass rod, moistened with the reagent, to a film of the dried substance upon a white porcelain surface. Sulphuric acid had no action. Nitric acid gave no reaction. The mixture of these two acids gave no action in the cold, but gave yellow color upon warming. Sulphuric acid and potassium dicbromate, applied as in the fading purple test for strychnine, gave *a fading red*, the color appearing *very distinctly*, for only a moment, fading quickly to blue, and then quickly and completely disappearing. I found 0.00033 grams of acetanilid to give this reaction distinctly. When manganese dioxide was substituted for the dichromate a red color was obtained, which lasted for a few moments and then completely disappeared. This last test is not the same given by berberine, inasmuch as the color produced by berberine is permanent, or, at least, lasts for a considerable length of time.

"Vitellis' atropine test is inapplicable to acetanilid, as the fuming nitric acid converts the acetanilid into nitro-phenyl-acetanilid, a brownish body, as before stated.

"The only impurity that is especially liable to occur in this drug, certainly if the articles from which it is made be pure, is that of aniline, slight traces of which should condemn the drug for medicinal use. The following test is given for its detection: *Triturate an excess of acetanilid with water to make a mixture, and add a solution of sodium hypobromite; if the product is pure the solution will remain yellow and limpid: but if it contains the slightest traces of aniline, a plentiful orange-red precipitate will be formed, the liquid assuming the same color. The only means of a quantitative estimation that I found is the following: †Nitrous anhydride is passed into a cooled solution of acetanilid in glacial acetic acid, and the green solution thus obtained is poured into cold water, when a yellow body, nitroso-acetanilid, $C_6H_5N(NO)C_2H_3O$, is precipitated. The reaction is said to be nearly quantitative, but the compound formed is a very unstable body, decomposing when heated with water, and exploding when heated without it. The process is altogether too cumbersome to be of any use practically, and a quantitative test for aniline is still wanted."

*Phar. Jour. Trans., (3), **17**, 685.

†Jour. Chem. Soc., **30**, 205.—By Otto Fischer—Ber., **9**, 463, 465.

THE OILS AND FATS.

Test for Adulterations in Oil of Almonds.—A. Kremel (Chem. Zeit. Rep. **13**, 46.) 15 gms. of almond oil are mixed with 2 cc. water and 2 cc. fuming nitric acid. After shaking, the solution is allowed to stand about 15 hours, when it separates to a white mass and a colorless liquid if the oil is pure. S. C.

Test for Oil of Bitter Almonds.—A. Kremel (Chem. Zeit. Rep. **13**, 46). If the natural oil of bitter almonds is heated with an excess of alcoholic solution of caustic potash, and then the excess of alkali neutralized with hydrochloric acid, a separation of benzoin takes place which is equal to 40 or 50% of the oil used. The artificial oil does not give this reaction when treated in the same manner. With this test also the oil from apricot kernels gives a small quantity of benzoin and the oil from the cherry laurel, which is identical with bitter almond oil, gives no benzoin.

S. C.

Densities and Refractive Indices of Certain Oils.—J. H. Long (Am. Chem. J. **10**, 392). Prof. Long has determined the specific gravities and refractive indices of olive, cotton-seed, sesame, mustard, peanut, castor and lard oils. The determinations were made with a Meyerstein spectrometer. For the results obtained the reader should consult the original paper.

Cotton-Seed Oil and Beef Fat in Lard.—J. A. Wilson, Chem. News, **59**, 99. It would seem that old samples of oils fail to give the reaction with silver nitrate. Experiments were made on the surface tension of oils, and it was found that pure dried melted lard, dropped on water of 100° F. does not extend or increase in size, while beef-fat or mutton-fat spreads over the surface, breaking up into innumerable little drops, and is agitated by a rapid circular motion. By mixing pure lard with different per cents of beef-fat or mutton-fat and allowing a drop to fall on the water and noticing the time and degree of expansion approximate determinations of beef-fat were made, but eight months after with the same lard, the drop gave results similar to beef-fat alone. The most accurate and delicate test for fat and oils in the author's experience is that of Hubl, with iodo-mercuric chloride.

A. H. W.

The Gravimetric Determination of Glycerine by Oxidation with Permanganate of Potash in Acid Solution.—H. Grünwald. Ztschr. angew. Chem. 1889, 34.

The author criticises the method of Planchon and improves upon it by retaining the carbonic acid in caustic potash solution, and the water by dry calcium chloride. The oxidation of glycerine by permanganate takes place according to the equation $C_3H_8O_3+7O=3CO_2+4H_2O$, and on this as a basis the author made twelve analyses with a mean error of 1.6%, on quantities varying from 0.2 gm. to 1.4 gms. J. E. W.

Estimation of Glycerine in Crude Glycerine.—F. Filsinger. Ztschr. angew. Chem. 1889, 3. The author gives results obtained by the use of the Acetin method of Benedict and Cantor and finds that with pure glycerine the results agree very well among themselves, but are somewhat lower than the specific gravity would would call for according to Gerlach's tables, while with soap glycerine the results are often 3.5% too high. J. E. W.

Adulteration of Lard with Cotton-Seed Oil.—A. Brijard and J. Waldbauer, Ztschr. angew. Chem. 1889, 119.

The authors review the methods of Bechi and Hübl for the detection of cotton-seed oil in lard; and compare the work of Williams and of Jones as to the absorption of iodine by this oil, with work of their own. (See this Journal, Vol. II, p. 275.) J. E. W.

The Bromine Absorption of Mineral Oils.—J. B. McArthur, J. Soc. Chem. Ind. **7**, 64. The bromine added was in solution in recently distilled carbon disulphide, and a known excess was added to a known weight of the oil also in solution in carbon disulphide. After allowing to stand for a certain length of time potassium iodide in aqueous solution is added and the iodine liberated determined with a decinormal solution of thiosulphate. The bromine solution was also approximately decinormal and was found to keep without rapid alteration when shielded from light and moisture. The author finds that the amount absorbed is generally increased in presence of moisture and by exposure to light; the amount absorbed is greater for a long than for a shorter time; it decreases as the solution is diluted, and increases with an increase in the excess of bromine present.

The Adulteration of Tallow with Cotton-Seed Oil.—R. Williams, J. Soc. Chem. Ind. **7**, 186. As much as 40 per cent of cotton-seed oil may be added to tallow without lowering the melting point greatly. A sample of genuine tallow melting at 110° F. melted at 102° F. after 40 per cent. of cotton-seed oil had been mixed with it.

The only method for detecting this adulteration of any value is the determination of the iodine absorption, which gives indications of great value; as little as 5% cotton-seed oil may be detected in this way. For all practical purposes, it is quite accurate enough to assert that every percentage of iodine absorbed in excess of that required by genuine tallow indicates adulteration with one and a half per cent. of cotton-seed oil. Pure tallow absorbs 40.8 per cent. iodine; pure cotton-seed oil 109.1 per cent.

The Rancidity of Fats.—Max Gröger, Ztschr. angew. Chem. 1889, 62. The author considers that fats become rancid by reason of the presence of water in the fat acids and glycerine, these being oxidized by the oxygen of the air. This oxidation must extend to the fat acids as well as to the glycerine, the latter not having been noticed in the free state. The fat acids break up into acids poor in carbon but rich in oxygen, belonging partly to the fat acid series and partly to the oxalic series, of which azelaic acid may be artificially prepared by the oxidation of the fat acids by nitric acid.

J. E. W.

The Estimation of Neutral Fat.—Max Gröger, Ztschr, angew. Chem. 1889, 61. The author finds that a large excess of half-normal alcoholic potash solution is necessary to completely saponify the neutral fats, and also that it is not only necessary to determine the percentage of neutral fat but also the equivalent weights, or the so called saponification numbers of the fats.

J. E. W.

A Modification of Koettstoefer's & Reichert's Methods for the Analysis of Butter and Other Fats.—W. F. Lowe, J. Soc. Chem. Ind. **7**, 185. "The method is a combination of Koettstoefer's and Reichert's methods, and is carried out in the following manner:—About 2 grams of the clarified fat is saponified with 10 cc. of normal alcoholic KHO in a flask tightly closed with a tied down cork. The alcohol is then boiled off on the

water bath, and the soap dissolved in 50 cc. of hot water. The excess of KHO is then titrated with $\frac{n}{2}$ H_2SO_4 solution, using one small drop phenol-pthalein as indicator. This gives the amount KHO required to saponify the total fatty acids, as in Koettstoefer's process; 25 cc. (altogether) of $\frac{n}{2}$ H_2SO_4 is then run in, two pieces of tobacco-pipe stem added, and the flask connected with a Leibig condenser and 50 cc. distilled over. The distillate is filtered and titrated with $\frac{n}{10}$ KHO solution, and phenol-pthalein as indicator.

This gives the volatile fatty acids as in Reichert's method, and also gives a second data for the constitution of the fat. A third data may be obtained by washing the fixed acids remaining in the flask, and adding to them any that have passed over into the distillate and been separated by filtration, and also any remaining attached to the tube of the condenser, dissolving them in alcohol and titrating with $\frac{n}{2}$ KHO and phenol-pthalein.

The same flask is used throughout the whole process, the fat being saponified, titrated, and distilled, in the one flask (one of Cubley and Preston's conical flasks of about 200 cc. capacity answers very well, and is sufficiently strong for the purpose). If the flask is tightly corked and heated on the water bath and well shaken, the saponification takes place almost at once, and ten minutes heating is quite sufficient for this amount of fat. The whole process (without the titration of the fixed acids) does not occupy more than two hours, and can be carried out even in less.

The standard alcoholic KHO is prepared with rectified spirit, as purified methylated spirit does not seem to answer so well.

"Blank experiments are made with each set of assays, two checks of 10 cc. of alcoholic KHO being boiled down and titrated with $\frac{n}{2}$ H_2SO_4. In measuring the alcoholic KHO it is very needful to allow the pipettes to drain for the same length of time. The writer allows them to drain for half a minute, and then touches the liquid with the point of the pipette. The whole of the volatile acids do not come over in 50 cc. of distillate, for if another 50 cc. of water be added to the flask and distilled a fresh quantity will come over, but is too small to be of importance. In an experiment tried in this way the second 50 cc. of distillate required only 0.15 cc. of $\frac{n}{10}$ KHO. An experiment was also made to see if any volatile acids (acetic, for instance) could be distilled over

from the alcoholic KHO; 10 cc. well heated in a closed flask in the same manner and for the same length of time as the fats, the alcohol boiled off, the residue dissolved in 50 cc. of water, 25 cc. of $\frac{n}{2}$ H_2SO_4 added, and 50 cc. distilled over. The distillate required only one drop (.05 cc.) of $\frac{n}{10}$ KHO to produce a permanent coloration with phenol-pthalein.

The following table gives some results obtained by the process. The results are expressed in milligrams of KHO required for 1 gram of fat."

	Total Fatty Acids. Milligrams of KHO required for 1 gr. of fat.	Volatile Fatty Acids. Milligrams of KHO required for 1 Gram of Fat.	Volatile Fatty Acids. Number of cc. of $\frac{N}{10}$ KHO required for 2.5 Grams of Fat.	Insoluble Fatty Acids. Milligrams of KHO required for 1 Gram of Fat.	Process.
Kiel butter	222	35.6	15.9		Combined Proc's
do.	227	36	16		do.
Butterine	190.4	4.2	1.8		do.
do.	192.6	4	1.7		do.
do.		4.1	1.8		Reichert's
do.		4.2	1.8		do.
Mutton suet	190.4	0.6	0.3		Combined Proc's
do.	189.9	0.6	0.3		do.
Kiel butter with 10.5 per cent butterine	224.9	28.5	12.7	199.9	do.
Kiel butter with 20.7 per cent. butterine	224	26.8	11.9	197.6	do.

TOXOCOLOGY.

Arsenic in Glass.—Marshall and Pott's (Am. Chem. J. **10**, 425). The old statement that the arsenious oxide added to the batch in glass making is all volatilized by melting is incorrect. The authors find in 5 gms. of glass (domestic and foreign) from 0.102 to 0.446 gms. As_2O_3. All the samples of clear glass examined, with one exception, contained arsenic. When a tube made of such glass is heated and pure hydrogen passed through it a brown coloration makes its appearance at the point in contact

with the flame.* The strong acids, ammonium hydroxide and ordinary reagents dissolve no arsenic when kept in such glass for long periods; but solutions of fixed alkalies dissolve a noticeable amount of arsenic—in one case a 10% solution of caustic soda was found to take up a perceptible amount in 69 hours and caustic potash of the same strength in 24 hours.

The authors find also that most of the commercial caustic soda (even that sold as chemically pure) contains arsenic. Of the samples examined but one sample, made from Solvay process soda, was free from it. Sodium carbonate sometimes contains arsenic and is sometimes free from it. Samples containing arsenic can be purified by several recrystallizations. Four samples of caustic potash were examined for arsenic, but found to be free from it.

E. H.

NOTES.

—Hazura finds that olive oil contains a notable amount of linolic acid, $C_{18}H_{32}O_2$, amounting to more than 7% of the oleic acid present; peanut oil also contains this acid. (Ztschr. angew. Chem. 1888, 696).

—König and Stutzer note the occurrence in commerce of artificial coffee beans made by moulding wheat flour and roasting the beans. According to Stutzer the fraud may be detected by throwing the beans into a strong, hot, oxidizing solution, (aqua-regia, or hydrochloric acid and potassium chlorate). The true bean is decolorized much more rapidly than the counterfeit. When the beans are thrown into ether, the coffee beans float upon the surface, while the artificial beans sink (See Ztschr. angew. Chem. 1888, 630, 699).

—The explosion of a cylinder of compressed gas in Dublin, Jan. 28, 1889, caused the death of Mr. T. A. Bewley. During the rush of the holidays, a red cylinder of oxygen was sent to fill an order, although the invariable rule had been to ship hydrogen in red cylinders and oxygen in black. This cylinder was afterwards returned and through mistake filled up with hydrogen before the oxygen was all expelled, and the mixed gas was ignited in

*Ztschr. anal. Chem. **22**, 400.

some unexplained way with a fatal result. Mr. W. N. Hartley suggests a precaution to avoid such accidents, namely, to make the fittings for hydrogen and oxygen cylinders so entirely different that it would be impossible to charge with the wrong gas (See Chem. News, **59**, 75). A. H. W.

—A monument to Dr. Joseph Priestly has been erected in the First Unitarian Church, Phila. It was unveiled and dedicated on March 13. An oration was delivered by Rev. C. C. Everett, D. D., Dean of the Theological School of Harvard University, and addresses upon Priestley's scientific services and attainments were delivered by Professors Joseph Leidy and J. P. Lesley.

—A new antiseptic soap is made by incorporating with the soap mass mercuric iodide in solution in potassium iodide. This soap has been tried in cases of eczema with marked success and in parasitic skin dieseases such as favus and ringworm, (See Jour. Soc. Chem Ind. **7**, 192).

PERSONALS.

—Mr. J. E. Whitfield of the U. S. Geological Survey has accepted a position with Booth Garrett & Blair, Phila.

—Mr. O. S. Doolittle has been appointed Chemist of the Philadelphia & Reading R. R. with headquarters at Reading, Pa.

OBITUARY.

—Dr. J.H. Kidder, late president of the Chemical Society of Washington: Dr. Jerome Henry Kidder was born near Baltimore Oct. 26, 1842. He graduated from Harvard College in 1862, and immediately thereafter entered the military service of the U. S. and served until the close of the war. In 1866 he entered the medical service of the Navy and 1876 reached the position of surgeon. Since 1874 he has been connected with the scientific service of the government, chiefly with the Fish Commission, of which at one time he was Assistant Commissioner. Since 1888 he has been connected with the Smithsonian Institution, and in that year was elected president of the Washington Chem. Society. Dr. Kidder's work in chemistry was chiefly of a medical nature, and in his retiring address in Feb., '89, he described the results of his observations on crystalline deposits on glass exposed to contamination

with expired air. In view of the recent publication of Berthelot's work on poisonous alkaloids in expired air, Dr. Kidders' address assumes unusual interest. Dr. Kidder was permanently identified with other scientific societies in Washington and was an honored and active member of the Cosmos Club.

He died of pneumonitis, after a short illness, on Monday, April 8th. H. W. W.

—Dr. Isidor Soyka, Professor of Hygiene in the German University at Prague, committed suicide by shooting himself on Feb. 23, 1889. S. C.

—Michel Eugene Chevreul, distinguished for his researches on the theory of colors and for his investigations on the fats, died in Paris, April 9, aged 102 years 8 months and 9 days. Chevreul has long been famous, but since he attained the age of 100 in 1886 others than chemists have felt an interest in his career. He was born at Angers, Aug. 31, 1786, and attended the chemical school there, afterwards studying with Vaquelin. He was successively Professor in the Lycee Charlemagne and in the Gobelin dyeworks. In 1824 he was elected member of the Institute and in 1834 Director of the Museum. So closes a long and useful life. We can do no better than follow his example in industry, modesty, temperateness. E. H.

NEW BOOKS.

AN ELEMENTARY TEXT-BOOK OF CHEMISTRY BY WM. G. MIXTER, PROFESSOR OF CHEMISTRY IN THE SHEFFIELD SCIENTIFIC SCHOOL OF YALE COLLEGE, NEW YORK: JOHN WILEY AND SONS. 12MO., 459 PP. PRICE $2.50.—This is an excellent book. The facts are well arranged and concisely and accurately stated, and the book is a safe guide as far as it goes. It may well be doubted, however, whether the book is fitted for use as a text-book for beginners—the use for which it is intended. It is certainly desirable in a text-book that the facts known and theories held shall be so presented as to make their comprehension easy for the student. Chemistry seems to be a difficult subject for most minds and it is by no means difficult for the teacher to bring about such a condition of things that the student despairs of compre-

hending and loses all interest in the study. One of the methods for bringing this about is to begin with a statement of the principles involved, and discuss at length the subjects of mass, temperature and heat, the balance (theory of), crystallography, the properties of gases, etc., etc. While this method may produce, as in this case, an excellent treatise, the resulting book is quite unfit for beginners. The problem is, not how much can we give them, but *how little will suffice;* such matters must be studied and understood, but they should be deferred until their acquisition is necessary for further progress.

On the other hand, some teachers proceed upon the idea that all the theories advanced must be evolved from a consideration of the experiments performed by the pupil. The result of such an attitude is seen in Shepard's text-book. It will be found by those who try this latter method, that while in bringing out some classes of phenomena the results are excellent. in other cases the results are disappointing—mainly because they are not commensurate with the time expended, and if carried too far it wearies the pupil. Some teachers advocate the historic method and begin the subject with a sketch of the history of chemistry. This method in skillful hands lends human interest and gives good results, but may easily be overdone. It is only by a happy blending of all these methods that the best results can be obtained. As far as is possible everything should be subordinated to awakening interest in the pupil—*for this means work on his part not to be secured in any other way.*

Prof. Mixter has erred mainly, we think, in arrangement. Historically, oxygen is the most important element, since modern chemistry dates from its discovery; it is also most important practically, because the most abundant in the free state and in its compounds. For convenience oxygen should come first, then perhaps Hydrogen, Nitrogen and Carbon, in the order named. To arrange the elements according to the periodic system, as Prof. Mixter has done, while it corresponds better with our knowledge, and is therefore more "scientific" is attended with numerous inconveniences. It would be more logical to treat the whole subject of organic chemistry under Carbon, as Eliot and Storer have done, but few teachers who have tried the plan will adhere to it because of the

inconvenience attending it. Another mistake occurs in the discussion of the subject of valence, which is taken up on p. 80. This is not only difficult to acquire so early, but most students fail to see its usefulness and scoff at it. It is better, perhaps, to postpone all mention of valence until the necessity for its study arises under the theory of salts in connection with nitric acid. A good part of this can then be taught without once mentioning the name "valence." The "Atomic Theory" is the last thing considered in the book and this is in our view still another mistake. As we proceed in our teaching upon the idea that the atomic theory represents acutal truth, nothing is gained, and much is lost by postponing its consideration. This subject may be treated very concisely, and should be taken up at the very beginning. It forms a proper introduction to the study and the only one needed. Nothing is gained by substituting the term combining weights for atomic weights, as Roscoe does, and as others must logically do unless this plan be followed; it is difficult to unlearn this term and the ideas associated with it:—The Atomic Theory may not be true; and if the teacher have doubts about this he can and should express them after the class has been told what the theory is.

As already indicated the mistakes in Prof. Mixter's book are mistakes in arrangement. The book is beautifully printed and illustrated and the cuts have a freshness that is pleasant to the eye accustomed to the use of the same plates in successive books for years past. E. H.

THE LIXIVIATION OF SILVER ORES WITH HYPOSULPHITE SOLUTIONS, WITH SPECIAL REFERENCE TO THE RUSSELL PROCESS, BY CARL A. STETEFELDT. THE SCIENTIFIC PUBLISHING CO. 8VO., 233 PAGES, $5.—This process was first introduced by Von Patera in 1858 and consisted originally in the treatment of chloridized ores with sodium thiosulphate (hyposulphite) which dissolved out the silver which was precipitated as sulphide. The defects of this process are: (1). That the perfect chlorination of the ore was difficult, especially with a calc spar gangue. (2). If lead and copper are contained in the ore they go into solution, partially or entirely, and are precipitated as sulphides with the silver. (3). The solution dissolves only the chloride contained in the

ore, leaving the metallic silver, polybasite, stephanite, etc. undissolved.

In the Russell process these defects have been overcome by the use of an extra solution consisting of a mixture of sodium thiosulphate and copper sulphate. This extra solution dissolves out the metallic silver as well as the asenical and antimonial silver sulphides. The lead is separated from the thiosulphate solution by the addition of sodium carbonate which precipitates it leaving the copper and silver in solution.

As compared with amalgamation the advantages are: That the ore need not be so finely crushed; the first cost of plant is less; less power is consumed; there is no expense for quicksilver in stock and no loss from this occurs; the percentage of silver extracted is usually higher; roasting need not be so carefully performed, and hence less salt may be used; the cost of chemicals is less than the loss of quicksilver in amalgamation; the lixiviation process permits the extraction of lead and copper as valuable by-products; it is less injurious to the health of the workmen; with gold bearing ores lixiviation extracts in many cases more gold than amalgamation. The disadvantages are, that greater chemical knowledge is required; the danger of loss by leakage and careless handling; the gold and silver are obtained as sulphides which are more costly to refine. The chemical reactions involved have been very carefully studied and are explained in detail. The practical details have also been well worked out and are carefully explained—in a word the book contains all the information necessary for the practical working of the process. The illustrations are numerous and well drawn. E. H.

ESTIMATION OF WATER FOR SANITARY AND TECHNICAL PURPOSES, BY HENRY LEFFMANN, M. D., PH. D., AND WILLIAM BEAM, M. A. P. BLAKISTON SON & CO., 12MO., 106 PP., $1.25.—This is a most excellent book. The authors take up first the History of Natural Waters, then Analytical Operations, Interpretation of Results and Analytical Data. Instead of a description of the reagents used a set of labels ready for use accompanies each copy of the book; the label itself indicates how the solution is to be made up. The book is an admirable digest of our present

knowledge and may safely be put into the hands of beginners. It is well illustrated and printed. E. H.

A LABORATORY GUIDE IN CHEMICAL ANALYSIS. BY DAVID O'BRINE, E. M., M. D., D. SC. SECOND EDITION, JOHN WILEY AND SONS. 8VO., 237 PP., PRICE $2.—The book is divided into eight chapters headed: How the Reagents are Made, their Uses and Tests; Tests in the Dry Way; Tests in the Wet Way; Separation of the Acids—Dry Way; Comparison of the Bases and Acids, Facts Hypotheses and Laws Employed in Chemistry; Water Analysis; Poisons, Ptomains, etc.; General Stoichiometry.

Treatises of this sort do not admit of much that is new in matter, but the scheme for Separation by Electrolysis on pages 123 and 124 may fairly be called a novelty. The book seems to be well planned and the details are well wrought out. E. H.

PAMPHELTS RECEIVED:—

—Notes of Work by Students of Practical Chemistry in the University of Virginia. No. XVII. Communicated by F. P. Dunnington.

—Notes on the Electrolytic Assay of Copper. By Wm. Glenn, Baltimore, Md.

—Michigan State Pharmaceutical Association. Proceedings Sixth Annual Meeting, 1888.

—On Auerlite and Sulphohalite, two new Minerals. By W. E. Hidden and J. B. Mackintosh.

—Regulations Concerning Analysis of Foods and Drugs in the District of Columbia, under control of the Commissioner of Internal Revenue.

—Report of the Commissioner of Internal Revenue for the Fiscal Year Ended June 30, 1888.

—The Mineralogy of Pennsylvania. Part I. By John Eyerman, Easton, Pa.

—On the Mineralogy of the French Creek Mines, in Pennsylvania. By John Eyerman.

—Infant Food and Infant Feeding. By Prof. Edgar Everhart. A paper read before The Texas State Geological and Scientific Association, Houston, Texas.

—Composition and Evaporative Power of Kansas Coals. By E. H. S. Bailey and L. I. Blake. From the 6th Biennial Report of Kansas State Board of Agriculture.

—University Studies. Published by the University of Nebraska, Lincoln, Neb. Vol. I. No. 2.

—Bulletins and Reports from the following Agricultural Experiment Stations have also been received: Conn., New Haven, Bulletins 96 and 97, and Annual Report for 1888.—Storrs School Station, Mansfield, Mass. Report for 1888 and Bulletins 32 and 33.—Kentucky, Lexington, Bulletins 16 and 17.—Illinois, Champaign, First Annual Report for 1887-88, and Bulletins 3 and 4.—Wisconsin, Madison, Bulletins, 17, 18 and 19.—Missouri, Columbia, Bulletins 4, 5 and 6.—Louisiana, Baton Rouge, Bulletins 18, 19, 20 and 21.

ERRATUM.

Vol. 3, p. 92, first and third lines *for* Anerlite *read* Auerlite.

VOL. III. [JULY, 1889.] PART 3.

THE

Journal of Analytical Chemistry.

DETERMINATION OF ASH IN MOLLASSES, HONEY, ETC.

BY H. W. WILEY AND HUBERT EDSON.

The use of metallic silver prepared by reducing the chloride with an alkaline solution of grape sugar or by direct reduction with iron or zinc has been recommended as an aid in the oxidation of the carbon in the preparation of the ash from sugars, molasses, etc. The metallic silver acts as a carrier of oxygen and after the completion of the reaction can be easily recovered from the ash by solution in nitric acid or otherwise.

Lucien* recommends the use of the oxide of zinc for this purpose. The oxide of zinc easily gives up its oxygen to the carbonaceous material and is again easily oxidized in the muffle where the incineration takes place. At the end of the process, therefore, the ash is burned perfectly free from carbon and the zinc remains in the state of oxide. The weight of the ash can therefore be easily determined by deducting the weight of the oxide of zinc which has been added. The amount of oxide of zinc used, as recommended by Lucien, is 50 mg. to 2½ g. of molasses or 5 g. of sugar. Platinum dishes were used and the platinum appeared to suffer no injurious effect from the action of the zinc.

We have given this method of ash determination a thorough trial and the results are given in the following tables:

*Bulletin de L'Association des Chimistes, Vol. 6, p. 356.

Experiments on the use of ZnO in Ash Determination.

Sample of "Third" Molasses from Calumet Plantation, La.

TABLE No. 1. No ZnO Used.

Date.	Weight Sample.	Weight Ash.	Per cent. Ash.	Remarks.
April 1	2.4614	.1983	8.06	Ash rather dark
" 1	3.4686	.2810	8.17	" " "
" 1	4.4886	.3602	8.02	" " "
" 3	2.4913	.1997	8.01	Ash very slightly dark
" 3	3.4904	.2785	7.98	" nearly white
" 3	4 4865	.3591	8.00	" rather dark
" 4	2.4877	.2014	8.10	" quite dark
" 4	3.4878	.2785	7.99	" " "
" 4	4.4931	.3596	8.00	" " "
			Mean ₰ 8.04	

TABLE No 2. ZnO Used.

Date.	Weight Sample	Weight Ash + ZnO.	Weight ZnO	Weight Ash.	Per cent. Ash.	Remarks.
April 1	2.4644	.2453	.05	.1953	7.97	Ash white, better in appearance than without ZnO.
" 1	3.4865	.3346	.05	.2846	8.17	Ash white, better in appearance than without ZnO.
" 1	4.4334	.4083	.05	.3583	8.08	Ash white, better in appearance than without ZnO.
" 3	2.4904	.2482	.05	.1982	7.96	Ash white, slightly better in appearance than without ZnO.
" 3	3.4955	.3462	.07	.2762	7.90	Ash white, nearly same in appearance as corresponding ash without ZnO.
" 3	4.4847	.4486	.09	.3586	8.00	Ash white, slightly better in appearance than without ZnO.
" 4	2.4926	.2496	.05	.1996	8.01	Ash very white, much better in appearance than without ZnO.
" 4	3.4840	.3425	.07	.2725	7.82	Ash very white, much better in appearance than without ZnO.
" 4	4.4805	.4496	.09	.3596	8.03	Ash very white, much better in appearance than without ZnO.
					Mean ₰ 7.99	

TABLE No. 3.

Date.	Loss ZnO.	Per Cent. Ash. Corrected.
April 1	.0015	8.21
" 1	.0013	8.11
" 1	.0030	8.08
" 3	.0032	7 99
" 3	.0032	8.07
" 3	.0016	8.07
" 4	.0021	7.88
" 4	.0028	8.09
" 4		
		Mean ₰ 8.06

First ash burned with ZnO April 4. The sample and ZnO were thoroughly mixed together with H_2O.

The second and third samples of same date were mixed with alcohol, evaporated on steam bath, and residue incinerated. The ashes in both cases much improved in appearance by this treatment.

As will be seen from the above table the mean percentage of ash in the samples of molasses used when burned in the muffle in the usual way was 8.04 per cent. In no case was the ash perfectly white. In the same samples in comparative determinations where oxide of zinc was used (Table No. 2.) the mean percentage of ash was 7.99. The ash in all cases was very much improved in appearance and apparently should have weighed much less than when no oxide of zinc was used. In the determinations made on the 4th of April the zinc oxide was thoroughly mixed with the molasses by means of water or alcohol, while in the determinations made previous to that date it was simply sprinkled over the mass in the dish. The samples of ash obtained by previous mixing of the oxide with water or alcohol were very much whiter than any of the others. In order to determine whether there would be any loss from the ignition of the zinc oxide alone at the temperature used, a series of determinations was made which showed an average loss of nearly 2 mg. for each 50 mg. of oxide employed. It is doubtful whether this loss takes place after the zinc oxide is thoroughly mixed with the sample, but in Table No. 3 the percentage of ash is given after correcting for the possible loss of zinc oxide. The mean percentage of ash as corrected in this way, would be 8.06.

It is seen by the above work that for quantitative determination the numbers obtained with and without the zinc oxide are so nearly identical as to render its use of no particular value. If the ash is to be analyzed quantitatively after it is obtained the presence of the zinc would probably be of more injury than the trace of carbon which would remain without its use. We are therefore of the opinion that the method in ordinary use of determining the ash directly by carefully incinerating in the muffle, is to be preferred to the methods depending upon the admixture of any other substance to be used as a carrier of oxygen.

IODINE ABSORPTION AS A TEST FOR THE PURITY OF ESSENTIAL OILS.*

BY H. W. SNOW.

In a hitherto unpublished paper presented to the faculty of the school of pharmacy of the University of Michigan, as a thesis (1884), the writer reported some experiments on the reaction which takes place between iodine, alcoholic solutions of mercuric chloride and turpentine by which the iodine is decolorized and partly at least taken up by the mercury to form iodide of mercury. At that time it was only the nature of the mercury salt which was under investigation. Some few weeks ago while engaged in the examination of a number of samples of oil of peppermint, the earlier work occurred to my mind and the thought suggested itself that the free manner in which turpentine absorbed the halogens under these conditions might be made the means of determining its presence in other essential oils. The use of iodine absorption as a means of detecting adulteration is not new inasmuch as Hubl has employed it† for the detection of adulteration in fixed oils and his process which the writer used has been favorably reported on by a number of chemists. The writer had performed a number of experiments in this direction when the work of A. B. Stevens‡ came to hand, in which that investigator points out that oil of peppermint absorbs iodine quite freely. Mr. Stevens' experiments were performed under somewhat different conditions from my own and yielded substantially the following results. (1). On adding known volumes of oil of peppermint to known volumes of decinormal solution of iodine made with potassium iodide and with a mixture of alcohol and water for a menstrum that the iodine is decolorized. (2). That on determining the excess of iodine by thiosulphate solution it was found that the reaction proceeded up to nearly ten minutes, beyond which time no further increase in the iodine taken up occurred. (3). That the presence of alcohol prevented in some

* From the New Idea, March, 1889.

† Allens Com. Organic Analysis, [1886]. Vol. II, page 48, from Jour. Chem. Soc., III, page 641.

‡ Proc. Amer. Phar. Ass., 1888-97. [Amer. Drug., Oct., 1888-192, and other English reprints.]

measure the absorption of iodine and that the amount of iodine decolorized increased as the alcohol decreased. (4.) That on adding known volumes of oil of peppermint to known volumes of decinormal solution of iodine in *aqueous* potassium iodide and shaking continuously for ten minutes and then estimating the excess of iodine with thiosulphate solution, that 1 cc. of oil decolorized 30 cc. of $\frac{n}{10}$ solution of iodine. (5.) That when these figures are calculated into parts by weight, 100 parts of oil absorb about 42 parts of iodine. In my own experiments I followed Hubl's process without modification, as I had already noticed in my earlier experiments that mercuric chloride facilitated the absorption of the halogens. The process is as follows:

Introduce into a tared flask having a capacity of about 60 cc., 0.100 to 0.250 grammes of the oil to be tested and obtain the exact weight taken. Dissolve in 10 cc. of chloroform and add from 10 to 40 cc. solution of iodine and mercuric chloride prepared as directed below, noting the exact amount introduced. The amount of iodine solution added must be such that after standing an hour or so the solution will have a distinct iodine color, as the iodine must always be in quite decided excess. If the first portions are decolorized more may be added. After the solution has stood from 40 to 48 hours add 5 to 10 cc. of strong solution of potassium iodide and about 125 to 150 cc. of water. Titrate at once with decinormal solution of sodium thiosulphate, adding towards the last freshly prepared starch solution as an indicator. Calculate results to the basis of the amount of iodine absorbed by 100 parts of oil. The solution of iodine is made by dissolving 25 grammes of iodine in 500 cc. of alcohol and 30 grammes of mercuric chloride in an equal amount of the same solvent. Mix the two solutions and after twelve hours filter. Inasmuch as the strength of this solution is subject to considerable alteration in strength on standing, it should always be determined at the same time the titrations are made of that added to the chloroform solution of the oil. It is essential that after diluting the solution with water it should be titrated at once, as the compounds formed are extremely unstable as a rule and on standing iodine soon begins to be liberated so that the solution again acquires purplish to blue tint. Some comparative experiments running from 3 to

48 hours show that about 40 hours must elapse before the absorption of iodine is complete, and sometimes then it does not seem to be so.

	TIME OF DIGESTION.					
	3 hours	6 hours	15 hours	24 hours	40 hours	48 hours
Oil Peppermint	21.1	22.8	24.5			
" "					179	179
" "					110	110
" "					120	126!
" Bergamot			300			345
" Orange Peel, Bitter			321			362
" Turpentine	291	333	345	353		397

The following figures show the results and relate to the number of parts of iodine which 100 parts of the oil will absorb in the time specified:

	TIME OF DIGESTION.		
	6 hours.	15 hours.	40 hours.
Oil Peppermint*	23.	24½	
" " (adulterated)	84.7!	74.1!	
" " "	88.3!	73.6!	
" "			179
" "			109
" "			67
" "			71
" "			106
" "			105
" " Japanese			64
Menthol	none		
Oil Bergamot			345
" Limes			289
" Lemon			343
" Orange (Bitter)			362
Methyl Salicylate (synthetic oil wintergreen)		3¾	
Oil Sweet Birch (sold as oil wintergreen)		3¾	
" Wintergreen (true)		1	
" Anise			121
" Coriander			385
" Caraway			233
" Thyme		210!	183!
Thymol		201!	181!
Oil Lavender			286
" Cloves			467
" Sassafras			166
" Pennyroyal			152
" Camphor			129
Camphor		2¾	
Oil Turpentine (fresh distilled)			397
" Erigeron			280
" Copaiba			250

*An unusually fine article purchased four or five years ago.

The writer's experiments thus far indicate that the iodine absorption of essential oils may yield figures having a considerable value in the determination of the purity of oils, but it is of course not likely that these figures are constants, except perhaps in a few instances, any more than the specific rotatory power, specific gravity, etc., are constant. There are also some anomalies which need clearing up and it is necessary that maximum, minimum and average equivalents should be determined by making estimates with a number of samples of the same oil and of unquestionable purity. The only direct application which the writer has yet made of it is in the detection of turpentine in oil of peppermint with a partial application to the detection of oil of copaiba, accounts of which are given below.

OIL OF PEPPERMINT.

EXAMINATION FOR ADULTERATION.*

BY H. W. SNOW.

Large buyers of oil of peppermint will doubtless, like the writer, welcome any addition to the methods, chemical or physical, which have for their object the testing of this oil for adulteration. For this reason the writer needs no apology for appearing as a contributor in this field of chemical literature. In the following paper, alcohol, the oils of camphor, turpentine and copaiba as adulterants are considered. In addition to these the oils of pennyroyal, Japanese peppermint and erigeron are named as falsifications. Probably, however, the most vicious contamination, whether wilfully added or the result of careless growing, is that of the so-called ragweed oil and other "weed" oils, many of which materially lower the quality of the oil even when present in very small quantities. Physical properties, especially when coupled with careful fractional distillation, afford the only means at present known for detecting the presence of some of these oils, though for others means are at hand which are of considerable value in determining their presence.

ALCOHOL

has been named as having been freely used as an adulterant, but the writer does not believe it to be so used to any considerable

* From the New Idea, March, 1889.

extent, though it is always a possible addition. Magenta makes a reliable test for its presence. This aniline product is insoluble in oil of peppermint free from alcohol and moisture, though most samples of the commercial oil will dissolve it in small proportion. In examining twenty samples of oil, only one was found not to dissolve the dye at all and two others took it up in very small proportion, the rest on warming dissolved noticeable quantities. If alcohol were added at all it would scarcely be in less than 5 per cent. quantities, and this amount is easily detected by shaking the oil with a few grains of the reagent for fifteen or twenty seconds: use no heat. With 5 per cent. or more of alcohol the oil will be colored a distinct red. Light tints may be disregarded and are probably due to the presence of small amounts of moisture. One or two trials on known material will quickly teach the analyst how much color is permissible and render the detection of any important quantity of alcohol an absolute certainty. In the examination of eighteen samples of oil only two (from the same house) were found to contain alcohol, and even these two contained it in small amounts probably added to aid in concealing some other adulterant as one of them afterwards (only one was examined) was found to contain turpentine.

OIL OF CAMPHOR.

The writer's experiments on this adulterant were confined to the trying of Mr. A. B. Stevens' nitric acid test.* According to Mr. Stevens, it is applied as follows: Place a drachm of nitric acid (Sp. Gr. 1.42) in a test tube and add one or two drops of the suspected oil, and after gentle agitation set aside for a few minutes. The mixture will be of a yellow color and if pure will remain so. If as low as 5 per cent. of oil of camphor be present the mixture will turn red in fifteen or twenty minutes. The writer can heartily endorse this test and in addition may offer a few remarks. A perfectly good quality of oil will change color but very little on this treatment and assumes a light straw yellow color, which becomes lighter if anything on standing, but in applying the test to commercial oils it will often be found that they will assume a reddish brown color. This is not to be mis-

* Proc. Amer. Phar. Ass., 1888, 97. Amer. Drug., Oct., 1888, 192 and in other English reprints.

taken for the color produced by oil of camphor. The reddish brown fades on standing two or three hours; but the color of oil of camphor, a distinct red, will remain for 30 or 40 hours and perhaps even longer. The reddish brown often obscures for a time the test for oil of camphor; but on allowing the test tube to stand over night brown will fade and leave the reddish color, if oil of camphor is present, distinct and unmistakable. This test will reveal with perfect ease as little as 5 per cent. of oil of camphor. Another advantage of it is that it can be applied for the detection of oil of camphor in other oils beside peppermint which give no color, but a little color or a fading color with nitric acid. In the examination of twenty samples of commercial oil of peppermint by this means, six were found adulterated with oil of camphor. Of these five were obtained from Michigan firms, one being from a jobbing house and one was obtained from a New York house.

OIL OF TURPENTINE

is generally spoken of as a probable adulterant of essential oils and is specifically mentioned as being added to oil of peppermint. Among other tests great stress has been laid on the *rotation of polarized light*, the *fulmination with iodine* and the solubility in an *aqueous alcohol*. With the specific rotatory power the writer has no experience, but nevertheless is justified in warning against too implicit a reliance in this test. Its positive results when coupled with fractional distillation may perhaps be and doubtless are trustworthy; but as to its negative results it must not be forgotten that *French oil of turpentine* rotates polarized light in the *same* direction as oil of peppermint, and according to a statement now before the writer nearly as far (—35°) as the lower average of peppermint oil. It is highly probable that this known fact of the difference between American and French turpentine has already been taken advantage of to nullify the optical test for turpentine in oil of peppermint.

AQUEOUS ALCOHOL.

Oil of peppermint is completely soluble in its own volume of 80 per cent. by weight alcohol, but on the other hand oil of turpentine is as completely insoluble in an equal volume of alcohol of this strength. This difference between these two oils has been

recommended as a means of detecting the latter when added to the former, but whoever first announced this test must certainly have overlooked the fact that the two oils are *mutual* solvents and that therefore their solubilities when in mixture will be greatly different from either of the oils when separate. This is such a common occurrence in solubilities that it is somewhat surprising it should not have been taken into consideration. As a matter of fact 80 per cent. alcohol is worthless as a test for the presence of turpentine in oil of peppermint. The usual test of equal volumes will overlook the presence of 30 per cent. and only begins to show the presence when 40 per cent. of turpentine has been added, and increasing the amount of dilute alcohol does not as one might suppose increase the exactness of the test. Further, when a little alcohol is also added with the adulterant, as we have reason to suspect is sometimes done, then 50 per cent. of oil of turpentine escapes notice by this test. Long before such amounts are reached the adulterant may be detected by the sense of smell. This test it will thus be seen is a *false reliance* and is doubtless a favorite with unscrupulous dealers in essential oils.

DISTILLATION WITH CHLORINATED

lime and water is mentioned for the purpose of directing attention to the fact that chloroform is yielded in small amounts and may be detected by filtering the distillate through a well wetted filter paper and applying the *isonitrile* test for chloroform. It was long ago recorded that turpentine yielded chloroform when thus treated, and in experimenting to determine whether this fact might be made a means of detecting turpentine in oil of peppermint it was noted that this latter oil also yielded chloroform.

IODINE ABSORPTION.

In the paper preceding this the writer has directed attention to the iodine absorption of essential oils as a means of determining their purity. The work is far from complete, and the writer has thus far only made a practical application of this property of oils in the case of the detection of oil of turpentine in oil of peppermint with a possible detection of oil of copaiba. The writer is scarcely in a position to fix the absolute variation of the iodine absorption for peppermint oil, but this is not essential to

success in its application. In its determination for eighteen commercial oils, a considerable number of which were known to be adulterated, a very considerable range was found, running between 24 parts and 179 parts of iodine to 100 parts of oil. Ordinarily it is found between 70 and 110. In one case a very satisfactory and high priced oil purchased some years ago and before sharp and unscrupulous competition had injured the quality of oil of peppermint showed an absorption of only 24. In another case an equivalent of 179 was found. But it yet remains to be seen whether this latter oil was pure or not and whether true oil of peppermint will run so high as this. Where considerable amounts of turpentine have been added, iodine absorption will indicate it when determined direct for the oil, but it is always safer to resort to fractional distillation and compare the iodine absorption of the fraction with that of the oil undisturbed by distillation. The more thorough the fractioning is done the better, and the small fractional distillation flasks of 4 oz. and 2 oz. capacity serve the purpose well. The following is a good procedure:

(A.)

From 4 ounces of oil fraction off a fourth. Take this fourth (1 ounce), and again fraction it, taking only one-eighth (1 dram). After allowing this latter fraction to stand until the drops of water which will usually be obtained have subsided, determine the iodine absorption and compare it with the figures obtained from the undisturbed oil. In the case of an oil free from turpentine we may expect to find the two figures not very far separated, with the probability that the iodine absorption for the fraction will be somewhat higher than for the original oil. In the presence of turpentine the iodine absorption for the fraction will be much higher, than for the natural undisturbed oil. Thus, in an oil whose iodine absorption was 105, when only one ounce was taken and fractioned, the iodine absorption was found to be 123, and when to another portion of this same oil 13 per cent. of turpentine was added and a one dram fraction from one ounce as in the previous case was determined for its iodine absorption, this was found to be 280. With the double fractioning suggested more perfect separation can be obtained and the result will correspondingly be more

secure when dealing with small amounts of turpentine. Also, experience to date justifies the statement that an oil yielding, from the fraction of one dram from one ounce, less than 125 for its iodine absorption is not very likely to be adulterated with turpentine. Only once thus far has the writer found an oil having a higher absorption in the original than this, and as already stated that oil is still open to question. If the oil exceeds 125 it is a suspect, and running over 185 may be discarded. With much turpentine present the fraction will have an absorptive power over 225. After careful fractional distillation the turpentine may often be detected by its odor. The absence of alcohol should first be proven.

OIL OF PENNYROYAL.

The only test for oil of pennyroyal which the writer has seen is the chloral sulphuric acid test. It has already been pointed out that this test is unreliable and the writer's work simply confirms that of previous investigators in this respect. Oil of peppermint alone gives too deep a color for it to be easy to distinguish the less marked olive green which oil of pennyroyal gives, and it is only when the amounts become considerable that it is easy to note its appearance, and even then the reaction does not seem to be uniform. The writer has not searched the commercial oils particularly for this adulterant, but casually noted its presence in two oils, one of which contained it in such quantities as to be distinctly noticeable from its odor.

OIL OF COPAIBA.

The iodine absorption of oil of copaiba as determined from a single sample was found to be 250. According to Fluckiger the boiling point of this oil is very high (245° C and over), and it was thought these two properties might be of service in its detection. An oil was taken and after determining its iodine absorption it was fractioned retaining a fourth in the retort: the same oil was then adulterated with about 20 per cent. of oil of copaiba and after fractioning in like manner the iodine absorption was determined for the residue. The three figures were 110, 129, 193, so that it will be seen that the oil of copaiba increases the absorptive power of the last fraction remaining in the retort, but its own absorptive power when perfectly pure is not sufficiently high to

make it increase this power very much over the variations which may perhaps be found to occur in the case of different samples of oil of peppermint. However, by comparing the iodine absorption of the undisturbed oil with that of the last one-fourth fraction remaining in the retort some data will be obtained which will be suggestive of the probable purity or sophistication of the oil as regards copaiba. In this connection it must be mentioned that in fractionally distilling for this last purpose a small retort is to be preferred to the regular fractional distillation flask, because the higher boiling point of the latter portions leads to the condensation and dropping back into the flask of the oil to an extent which brings the distillation to a close.

CHLOROFORM SOLUTION OF CHLORIDE OF GOLD.

In their deportment towards a chloroform solution of chloride of gold, the essential oils show in some cases differences so far as tried. When only two or three drops of oil are boiled with 5 cc. of a 1 in 200 solution, oil of turpentine reduces the solution very quickly and with little or no color. Spangles of gold are seen to adhere to the side and bottom of the tube. Oil of camphor acts but little and only slowly developing a faint blue to violet color. Oil of Japanese peppermint gives no color or reduction. Oil of pennyroyal reduces it a little, giving dark color somewhat greenish. Oil of peppermint on the other hand reduces the solution quickly, giving first a brown then beautiful brownish red, gradually passing into a violet red with cloudiness. Oil of copaiba reduces the solution somewhat slowly, but on standing a few moments yields a beautiful blue solution which gradually fades. Some experiments were undertaken to see how far this might be applied to the detection of oil of copaiba in oil of peppermint. It must be confessed at the start that the indications are not so encouraging as they might be, but they were such as to justify a mention of the results. With considerable amounts of oil of copaiba present (20 per cent. or more), there is little or no difficulty experienced in noticing its presence, especially when compared with an oil of known purity beside it. The color of the solution is violet with marked tendency to the blue. With amounts of only about 10 per cent. however, the color approaches rather too nearly that afforded by pure oil of peppermint to make

the distinction a close one. In the latter case it is indispensable that two or three samples of oil of peppermint of known purity should be at hand to compare with the suspected samples, experiments being made parallel with and under the same conditions as those on the oil under investigation.

This color test like most of its class unless used with caution is apt to mislead, and with small amounts of adulterant the judgment drawn from its indications should be qualified. By its means it is easier and safer to demonstrate the absence of oil of copaiba than its presence. Only five oils were examined by this test, four of which were certainly free from the adulterant. The fifth gave indications of its presence, but was also known to be heavily adulterated with oil of camphor so that it is possible that the combination gave a test simulating that afforded by oil of copaiba.

THE ELECTROLYTIC METHOD APPLIED TO MERCURY. —SEPARATION FROM COPPER.*

BY EDGAR F. SMITH AND LEE K. FRANKEL.

The electrolytic precipitation of mercury has been successfully performed by different chemists; thus, J. B. Hannay (*Berichte d. d. chem. Gesellschaft*, **6**, 270) recommends a solution of mercury sulphate for this purpose, but gives no quantitative results. F. W. Clarke (*Am. Journal of Science*, **16**, 200; *Zeitschrift für analyt. Chemie*, **18**, 103) separated mercury from the solution of its chloride, freely acidulated with sulphuric acid, by using a current obtained from six Bunsen chromic acid ceils. Classen and Ludwig (*Berichte d. d. chem. Gesellschaft*, **19**, 323) employed a mercury solution, rendered slightly acid with nitric acid, and a current affording .5–1.0 cc. OH gas per minute. The time occupied in the deposition, at ordinary temperatures, was twelve to sixteen hours. Hoskinson (*Am. Chem. Journal*, **8**, 209) obtained good results with the nitrate. Smith and Knerr (*Am. Chem. Journal*, **8**, 209) employed a nitrate solution, in which there was considerable acid in excess, and with a current yielding 4 cc. OH gas per minute deposited .1 gramme of mercury in forty-five minutes. In all of these methods, which permit of the separation of mercury

*Read at the Stated Meeting of the Chemical Section of the Franklin Institute, April 16, 1889.

from many other metals, the mercury is thrown out as a mirror-like deposit, and drops of metal are plainly discernible.

We have observed that this metal can be separated without difficulty from solutions containing a large excess of alkaline cyanide, and with a comparatively weak current. The solution used was the chloride. The conditions, time, strength of current and results are as follows, with mercury alone:

Solution Contained Hg.	Found Hg.	Per cent. Difference.	Quantity of KCN.	Total Dilution.	Time in Hours.	Current in cc. OH Gas per Minute.
Grammes.			*Grammes.*	cc.		cc.
.1945	.1953	+.41	.26	175	16	.2
...	.1948	+.14	.26	...	16	.2
..	.1946	+.05	1.30	...	24	...
...	.1930	−.77	1.30	...	24	...
...	.1945	...	1.30	...	12	.2
...	.1945	...	1.30	...	12	...
...	.1945	..	2.60	...	12	.2
...	.1944	−.05	.65	...	12	...
...	.1942	−.14	.65	...	12	.2
...	.1956	+.56	.65	...	12	.2
...	.1957	+.61	1.30	...	12	...
...	.1948	+.14	1.30	..	12	.2

The deposits in the determinations were compact, rather gray in color, and showed in a few cases the drop-like nature so characteristic of mercury.

For washing purposes it was found best to use water only, because when this was followed with alcohol, we noticed that the latter detached thin films of metal, causing a loss in consequence.

The heat of the hand is sufficient to dry the deposit, though a warm iron plate may be used. Some of the deposits were allowed to dry slowly over sulphuric acid. In our experience, with about a hundred mercury depositions, we observed but five in which there was a slight oxidation of the metal. This only occured when a film of water remained in contact for some time with the deposited mercury.

In working with copper solutions, under conditions similar to those mentioned above, we discovered that this metal would not separate until the alkaline cyanide was completely decomposed. We, therefore, undertook a series of experiments with solutions containing both mercury and copper, hoping to effect their electrolytic separation. This seemed advisable, as the attempts in this direction had thus far not been as successful as might be de-

sired (See Luckow, *Zeitschrift für analyt. Chemie*, **8**, 24, and Classen, *Berichte d. d. chem. Gesellschaft*, **17**, 2,467; also *Zeitschrift für analyt. Chemie*, **24**, 247).

Our mercury solution contained the same quantity of metal as in the first experiments. Seventy per cent. of copper was added in each case. The quantity of cyanide varied from .65 grammes to 3.9 grammes; total dilution was 200 cc., and the current strength varied from .1 to .4 cc. OH gas per minute. Time, six to eighteen hours. Ten experiments were performed, and notwithstanding the mercury was completely deposited, slight quantities of copper were likewise thrown out of solution.

Thirty additional experiments were made; in each the quantity of mercury was .1945 grammes, while the copper varied from fourteen to seventy per cent. The quantity of alkaline cyanide varied from 3.9 to 8.5 grammes; total dilution remained 200 cc. The current averaged .4 cc. to .12 cc. OH gas per minute. Time, eighteen hours. The best results were these:

Hg Taken	Hg Found.	Difference in Per Cent.
Grammes.		*Per Cent.*
.1945	.1932	— .66
. . . .	.1920	—1.28
. . . .	.1927	— .92
. . . .	.1936	— .46
. . . .	.1930	— .77
. . . .	.1936	— .46
. . . .	.1954	+ .46
. . . .	.1938	— .36
. . . .	.1923	—1.13
. . . .	.1921	—1.23

These, as well as the results obtained in twenty experiments made later, seem to indicate that the presence of the copper exercises considerable influence upon the precipitation of the mercury —retards it. This fact, we think, was confirmed by ten or more experiments, in all of which a slight quantity of the mercury was retained in solution, notwithstanding the current was increased to 1.2 cc. OH gas per minute, and the amount of cyanide considerably reduced. The time of each precipitation was continued through seventeen hours.

Our attention was next directed to ascertaining whether or not a stronger current might be used to throw out the mercury, and

at the same time leave the copper in solution. The results with mercury alone were

Hg Present	Hg Found.	Difference in Per Cent.	KCN in Grammes.	Total Dilution.	Current in cc. OH Gas per Minute.	Time in Hours.
			Grammes.	*cc.*	*cc.*	
	.1827	—.27	1.5	200	2.8	16
.1833	.1832	—.05	1.5	200	2.8	16
	.1834	+.05	1.5	200	2.8	16

Using the same quantity of mercury and varying amounts of copper the results were as follows:

Hg Present	Per Cent of Cu Present.	Hg Found	Difference in Per Cent.	KCN in Grammes.	Total Dilution.	Current in cc. OH Gas	Time in Hours.
	Per Cent.		*Per Cent.*	*Grammes.*	*cc.*	3.2 cc. to 4 cc. OH per minute.	16
.1833	1	.1821	—.65	1.5	200		. . .
. .	10	.1828	—.27	. . .	. . .		. . .
. .	1	.1821	—.65	. . .	. . .		. . .
. .	14	.1833	. . .	. . .	. . .		. .
. .	10	.1821	—.65	. . .	. . .		. . .
. .	5	.1819	—.76	. . .	. . .		. . .
. .	3	.1815	—.98	. . .	. . .		. . .
. .	5	.1834	+.05	. . .	. . .		. . .
. .	3	.1836	+.16	. . .	. . .		. . .

which are recorded in the order obtained, and while the differences in percentage from that required vary from +.05 per cent. to —.98 per cent., and in six cases out of nine show a deficiency in the mercury, yet a careful qualtitative examination of each filtrate failed to detect this metal; the error must therefore be ascribed to other causes. It may not be improper to add that these determinations and separations were not always conducted in the same platinum vessels, and, further, those used by us ranged in weight from sixty-one to 135 grammes.

Where the quantity of copper exceeded twenty per cent. of the mercury, the results were unsatisfactory.

It is well known that silver can be separated qualtitatively from its cyanide solution, but whether its separation from copper could be effected in the same manner as that by which mercury and copper were separated, was undetermined; to ascertain this, we made forty experiments; ten of which resulted as follows:

Ag Present	Copper.	Ag Found.	Difference in Per Cent.	KCN in Grammes.	Current in OH Gas	Time in Hours	Dilution.	
	Per Cent		Per Cent.	Grammes.	cc.			
	10	.1257	+2.78		1		200	Copper precipitated with silver.
	10	.1299	+6.20		1		200	
	10	.1225	+ .16		1		200	
.1223	10	.1263	+3.20	.5	1	5	200	
	10	.1257	+2.78	to	1	to	200	
	10	.1244	+1.71	1.0	1	20	200	
	10	.1251	+2.20		1		200	
	10	.1242	+1.55		1		200	

Later, we diminished the quantity of copper with no better result. On increasing the quantity of cyanide, and also the strength of the current, the silver was very notably retarded in its deposition. After carefully repeating the work with no better outcome, we feel justified in saying that the cyanide method cannot be applied in the electrolytic separation of these two metals.

The current employed by us was obtained from storage batteries of the Julien form. Each cell contains nineteen plates, each of which is five and three-quarter inches square. In a long experience, with almost every form of battery, in electrolytic work, we have not had the same even, steady current for a series of hours as with the Julien form, and recommend it to all engaged in similar experiments. See further *Berichte d. d. chem. Gesellschaft*, **21**, 2892.

UNIVERSITY OF PENNSYLVANIA,
PHILADELPHIA, APRIL 13, 1889.

ON THE DETERMINATION OF THE ORGANIC NITROGEN IN NATURAL WATERS BY THE KJELDAHL METHOD.*

BY THOMAS M. DROWN, M.D., AND HENRY MARTIN, S.B.

The justification for the almost universal practice of determining the organic nitrogen in waters in the form of albuminoid ammonia is to be found in the great simplicity of the Wanklyn process. The amount of nitrogen obtained in the form of ammonia by the action of alkaline permanganate on nitrogenous organic matter in water bears no known relation to the total

* From the *Technology Quarterly*, vol. ii., No. 3.

nitrogen present, and chemists report widely differing results in consequence of differing practice. In the case of colorless waters, with small amount of organic matter, the differences are not usually great, but in most surface waters, with considerable organic matter in suspension and solution, the results often differ fifty or one hundred per cent. Some water analysts stop the distillation for albuminoid ammonia when 150 cc. have distilled over; others continue until 250 cc. are obtained, and again, others, strive to get all the ammonia they can by the addition of more alkaline permanganate, or by replacing the water which has distilled over.

There can be no doubt that the determination of the total organic nitrogen would be generally practised in place of the determination of the albuminoid ammonia, if there was available a short, easily executed, and accurate method for this purpose.

As the result of a very large number of experiments with the Kjeldahl nitrogen process, we think it may be safely said that this method leaves little, if anything, to be desired in these respects.

The modifications of the process, as usually practised, to adapt it to the determination of organic nitrogen in waters, are in the direction of its simplification. Thus the amount of organic matter in water is ordinarily so very small that the use of solid potassium permanganate for the final and complete oxidation of the organic matter is probably never necessary. In most of the analyses which we have made, we have continued to use it as a matter of precaution, but in the comparative experiments, with and without its use, the results have not differed. The volumetric determination of the ammonia by titration with very dilute standard acid was found to be less accurate and less convenient than the Nesslerisation of the distillate, in the usual method employed in water analysis. Further, the same flask is used for both the digestion and the distillation.

Professor W. R. Nichols experimented with the Kjeldahl process for the determination of the nitrogen in sewage with satisfactory results,* but the process has not yet, as far as we know, been used in regular water analysis.

The examination is conducted as follows:—500 cc. of the

* *Franklin Institute Journal*, August, 1885.

water is poured into a round-bottom flask, of about 900 cc. capacity, and boiled until 200 cc. have been distilled off. The "free ammonia" which is thus expelled may, if desired, be determined by connecting the flask with a condenser. To the remaining water in the flask is added, after cooling, 10 cc. of pure concentrated sulphuric acid.* After shaking, the flask is placed in an inclined position on wire gauze, on a ring-stand, or other convenient support, and boiled cautiously, in a good-drawing hood, until all the water is driven off and the concentrated sulphuric acid is white or a very pale yellow. The flask is then removed from the flame, and a very little powdered potassium permanganate added until, on shaking, the liquid acquires a green color, showing that an excess of the permanganate has been added. Should the color be purple instead of green, it shows that the water has not all been driven off. After cooling, 200 cc. of water free from ammonia are added, the neck of the flask being washed free from acid, and then 100 cc. of sodium hydrate† solution. The flask is immediately connected with the condenser, and then shaken to mix the contents.

The distillation at the start is conducted rather slowly, and the first 50 cc. are condensed in very dilute hydrochloric acid.‡ The contents of the flask may then be boiled more rapidly until 150 cc. to 175 cc. have altogether been collected. The total distillate is made up to 250 cc. with water free from ammonia, well mixed, and 50 cc. taken for Nesslerisation. No serious difficulty has been encountered from bumping when boiling the alkaline solution. The use of metallic zinc in the flask to facilitate the boiling is, of course, inadmissible, on account of the reduction of nitrates and nitrites, should they be present, to ammonia.

Any efficient condensing arrangement may be used for the collection of the ammonia. We have used with great satisfaction

* It is necessary to have for this purpose sulphuric acid which is very nearly, if not quite, free from nitrogen in any form. Baker & Adamson, of Easton, Penn., make an acid for this purpose which contains only 0.005 mgrm. of ammonia in 10 cc.

† The sodium hydrate solution is made by dissolving 200 gms. of commercial caustic soda of good quality in 1.25 liters of distilled water, adding 2 gms. of potassium permanganate, and boiling down to somewhat less than a liter. When cold, the solution is made up to a liter. The addition of the permanganate is to oxidise any organic matter which may be present in the caustic soda.

‡ The acid should be free from ammonia; 1 cc. of the acid is equivalent to 0.5 mgrm. of ammonia.

the condenser devised by Prof. S. W. Johnson, of the Connecticut Agricultural Experiment Station, which is described in *Bulletin* No. 12 of the United States Department of Agriculture, Division of Chemistry. It consists of a copper tank, 20 inches high, 32 inches long, 3 inches across the bottom, widening to 6 inches at the top. The tank is provided with an adequate supply of running water, entering at the bottom, and accommodates 6 or 7 block-tin condensing-tubes, three-eights inch internal diameter, which enter the tank through holes in the front side near the top, above the level of the overflow, and pass down vertically through the tank and out through rubber stoppers tightly fitted into holes in the bottom. They project about 2 inches below the tank, and are connected by means of rubber tubes to straight glass calcium chloride tubes with a bulb at the upper end. These glass tubes dip into 250 cc. flasks which receive the distillate. The distilling-flasks are connected with the tin condensing-tubes by means of rubber stoppers which carry a bulbed glass tube bent at right angles. This tube and its rubber stopper remain permanently connected with the tin tube.

The flasks rest on iron rings, and are heated with the free flame of a Bunsen burner. They should be carefully selected as to size and height. And the fixtures should be so arranged that all parts are interchangeable. There is then never any difficulty in putting the flasks promptly into place and connecting them with the condenser.

It is a good plan to have flasks, partly filled with water free from ammonia, connected with the condensing-tubes when not in use.

Before beginning a determination the water in the flask is boiled until the distillate shows, on Nesslerisation, that the apparatus is completely free from ammonia. Into the flask which receives the distillate there is put 1 cc. of the dilute hydrochloric acid and 50 cc. of water. The delivery tube dips into this liquid only during the collection of the first 50 cc. of the distillate. The flask is then lowered so that the tube remains above the liquid for the remaining time of the distillation.

In carrying out the operation, the most scrupulous care must be observed in preventing access of ammonia from any source.

The acid solutions will absorb ammonia from the air of the laboratory or from the dust of the room if they are allowed to remain uncovered for any length of time. This source of error has been found at times to be very large; quite enough to render a determination valueless. One experiment gave a gain of ammonia in twenty hours, by leaving the flask which contained the concentrated sulphuric acid uncovered, equivalent to 0.5 cc. of the standard ammonium chloride solution, and at another time the gain was 3 cc.

The operation should therefore be carried out without interruption, and for every determination, or set of determinations, a blank analysis with ammonia-free water should be made for a correction for the ammonia in the reagents and that accidentally introduced in the process.

We have not found that the presence of nitrates and nitrites in waters interferes with the accurate determination of the organic nitrogen. The error which has been found by Kjeldahl and Warrington* to be caused by nitrates in the determination of organic nitrogen seems to disappear under the conditions of great dilution which we have in natural waters.

The following experiments bear on this point:

1500 cc. standard ammonium chloride solution (=15 mgms. NH_3) and 10 cc. of standard potassium nitrite solution (=1 mgm. N) were boiled with 10 cc. of sulphuric acid, and the vapors condensed. This distillate contained only 0.15 mgm. nitrogen as ammonia and nitrous acid. The residue in the flask was made alkaline and distilled, and the ammonia obtained was precisely the amount taken, namely, 1500 cc. The experiment was repeated, using a nitrate in place of the nitrite, and under the same conditions the 1500 cc. of ammonia were recovered.

When a smaller amount of ammonia was used, we still failed to observe any loss. Thus when 10 cc. standard ammonium chloride solution and 10 cc. potassium nitrite solution (=0.1 mgm. N) were treated as above, there was obtained ammonia equivalent to 10.5 cc. Another experiment with the same quantities gave precisely 10 cc. of ammonia regained. With potassium nitrate instead of nitrite, in the same proportions as in the foregoing experiment, 10.5 cc. of ammonia were obtained.

* Chemical News, lii., p. 162.

The attempt to collect all the nitrous and nitric acids in the distillate was unsuccessful. The flask and condenser were connected by ground joints, so that the distillation could be continued after the sulphuric was concentrated. Owing, probably, to the shape of the flask, the acids were condensed, in part, in the neck of the flask, for after a second and a third addition of water and a renewal of the distillation more nitrous and nitric acids were obtained in the distillate. To see whether there was any loss of ammonia from ammonium sulphate in presence of sulphurous acid from the action of sulphuric acid on carbonaceous matter, the following experiments were tried: 10 cc. of standard ammonium chloride solution were treated with 10 cc of sulphuric acid and a weighed amount of Swedish filter-paper. Deducting for the nitrogen in the paper, determined in a blank analysis, 10.5 cc. of ammonia were obtained. In a duplicate experiment the ammonia obtained was 10 cc.—precisely the amount taken. In still another experiment with 50 cc. of standard ammonium chloride solution and filter-paper, 51 cc. were obtained.

No attempt has been made to compare, as regard the results obtained, the above-described method of determining the organic nitrogen with the combustion method of Frankland and Armstrong. But the following experiments on very dilute solutions of organic substances of known composition may serve to confirm the accuracy of the method.

One hundred mgrms. of pure crystals of urea were dissolved in one liter of water free from ammonia. Ten cc. of this solution were added to 500 cc. of water, and the analysis as above described. There was obtained 0.494 mgm. the theoretical amount being 0.466. In a duplicate experiment no solid potassium permanganate was added to complete the oxidation, and the result was 0.486 mgm.

A solution of uric acid in dilute potassium hydrate was made of the same strength as the urea, and 10 cc. taken for analysis. There was found 0.326 mgm. of nitrogen; required, 0.333 mgm. In a duplicate experiment without the permanganate, precisely the same amount was obtained.

A solution of naphthylamine, 100 mgms. to the liter, was dissolved by the aid of dilute hydrochloric acid. An analysis of 10

cc. gave 0.082 mgm. nitrogen; required, 0.097. Here again the result obtained by the omission of the permanganate agreed precisely with the determination in which the permanganate was used.

The following series of experiments, made on a sample of Cochituate water, shows the very close agreement of results in the determination of organic nitrogen under varying conditions. From a large bottle of water freshly drawn from the tap were taken five portions of 500 cc. each.

The first portion was treated by the usual method, already described.

The second portion was treated like the first, except that the addition of solid permanganate was omitted.

The third portion received 10 cc. of standard potassium nitrate solution, equivalent to 0.1 mgm. of nitrogen.

The fourth portion received 1 cc. of potassium nitrate solution, equivalent to 0.01 mgm. of nitrogen.

The fifth portion contained ten times the amount of potassium nitrite as the fourth portion, or 0.1 mgm.

The third, fourth, and fifth portions were treated in all respects like the first portion.

The results, expressed in organic nitrogen, parts per 100,000, were—

No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
0.0354	0.0354	0.0354	0.0365	0.0365

It may be of interest to give a few of the results obtained in the determination of the organic nitrogen by this method in natural waters in comparison with the results obtained as albuminoid ammonia. In the Table following will be found the analyses of some Massachusetts waters, during three successive months, June, July, and August, 1888.

In the first column is a record of the color, based on a comparison with the Nesslerisation of a known amount of ammonium chloride. Thus a color 1 means that the water possessed a color corresponding in depth and tint to 1 cc. of standard ammonium chloride solution (= 0.00001 gm. NH_3) when Nesslerised; a color

of 2 is twice the depth of color, and 0.1 is one-tenth the color of one. In the second column is the albuminoid ammonia obtained by the adding 40 cc. of alkaline permanganate to the water in the flask, after boiling off 150 cc. for free ammonia, and then distilling over 250 cc. In the third column is the organic nitrogen determined by the Kjeldahl process, converted into ammonia to make the figures comparable with the second column. All the determinations were made on the waters filtered through paper in the laboratory.

COMPARISON OF ALBUMINOID AMMONIA AND ORGANIC NITROGEN IN NATURAL WATERS.

I. *Surface Waters.*

Water.	Color.	Albuminoid ammonia.	Organic nitrogen calculated as ammonia.
Arlington, Reservoir—			
June	0.8	0.0256	0.0560
July	0.3	0.0274	0.0560
Boston Supply, Basin 4—			
June	1.0	0.0226	0.0470
July	0.7	0.0234	0.0400
August	0.9	0.0254	0.0460
Boston Supply, Basin 2—			
June	1.4	0.0350	0.0590
July	1.3	0.0294	0.0450
August	0.85	0.0234	0.0440
Boston Supply, Basin 3—			
June	1.9	0.0392	0.0790
July	1.7	0.0336	0.0540
August	0.8	0.0278	0.0590
Boston Supply, Lake Cochituate—			
June	0.35	0.0176	0.0420
July	0.10	0.0192	0.0390
August	0.10	0.0196	0.0420
Boston Supply, Mystic Lake—			
June	0.20	0.0252	0.0590
July	0.10	0.0244	0.0560
August	0.15	0.0212	0.0420
Bridgewater, Taunton River—			
June	2.3	0.0248	0.0520
July	1.0	0.0210	0.0440
August	0.7	0.0212	0.0390
Brockton, Reservoir—			
June	0.9	0.0234	0.0570
July	0.9	0.0260	0.0490
August	0.7	0.0310	0.0610

Water.	Color.	Albuminoid ammonia.	Organic nitrogen calculated as ammonia.
Brookline, Charles River—			
June	1.20	0.0320	0.0520
July	0.60	0.0216	0.0470
August	0.45	0.0208	0.0380
Cambridge, Fresh Pond—			
June	0.20	0.0162	0.0370
July	0.15	0.0170	0.0390
August	0.0	0.0172	0.0390
Clinton, Nashua River—			
June	0.1	0.0072	0.0230
July	0.2	0.0144	0.0360
August	0.2	0.0124	0.0320
Fitchburg, Reservoir—			
June	0.1	0.0124	0.0250
July	0.1	0.0158	0.0340
August	0.1	0.0146	0.0240
Great Barrington, Housatonic River—			
June	0.1	0.0122	0.0320
July	0.1	0.0120	0.0270
August	0.0	0.0134	0.0320
Nashua, Merrimac River—			
June	0.4	0.0120	0.0320
July	0.2	0.0132	0.0290
August	0.2	0.0106	0.0290
Lowell, Merrimac River—			
June	0.30	0.0142	0.0420
July	0.10	0.0148	0.0280
August	0.25	0.0134	0.0320
Haverhill, Merrimac River—			
June	0.40	0.0164	0.0490
July	0.20	0.0170	0.0420
August	0.20	0.0163	0.0348
Lake Winnepesaukee—			
June	0.0	0.0090	0.0230
July	0.0	0.0082	0.0190
August	0.0	0.0080	0.0200
Hyde Park, Neponset River—			
June	0.6	0.0286	0.0520
July	0.8	0.0432	0.1100
Lynn, Birch Pond—			
June	0.45	0.0194	0.0420
July	0.35	0.0216	0.0410
August	0.25	0.0174	0.0410
Malden, Spot Pond—			
June	0.3	0.0216	0.0440
July	0.2	0.0198	0.0390
August	0.2	0.0216	0.0390

Water.	Color.	Albuminoid ammonia	Organic nitrogen calculated as ammonia.
Montague, Connecticut River—			
June	0.15	0.0120	0.0220
July	0.20	0.0118	0.0250
Springfield, Connecticut River—			
June	0.4	0.0132	0.0390
July	0.15	0.0146	0.0320
August	0.15	0.0170	0.0380
New Bedford, Acushnet River—			
June	2.3	0.0296	0.0540
July	1.8	0.0278	0.0540
Northboro, Assabet River—			
June	0.7	0.0192	0.0490
July	0.4	0.0216	0.0380
August	0.4	0.0174	0.0410
Salem, Wenham Lake—			
June	0.10	0.0132	0.0320
July	0.05	0.0118	0.0290
August	0.00	0.0130	0.0260
Springfield, Ludlow Reservoir—			
June	0.10	0.0222	0.0460
	0.20	0.0214	0.0440
July	0.10	0.0220	0.0420
August	0.05	0.0198	0.0390
	0.10	0.0206	0.0370
Wayland, Reservoir—			
June	1.5	0.0272	0.0590
July	0.9	0.0364	0.0660
August	0.4	0.0244	0.0540
Whitman, Hobart's Pond—			
June	1.7	0.0538	0.0990
July	1.5	0.0426	0.0990
August	0.9	0.0342	0.0740
Wilmington, Shawsheen River—			
June	0.8	0.0242	0.0640
July	0.15	0.0114	0.0370
August	1.00	0.0316	0.0570
Woburn, Horn Pond—			
June	0.35	0.0290	0.0570
July	0.25	0.0324	0.0590
August	0.10	0.0240	0.0440
Worcester, Blackstone River—			
June	—	0.0350	0.1350
July	—	0.0440	0.1550
August	—	0.0830	0.3800

II. *Ground Water.*

(Without Color).

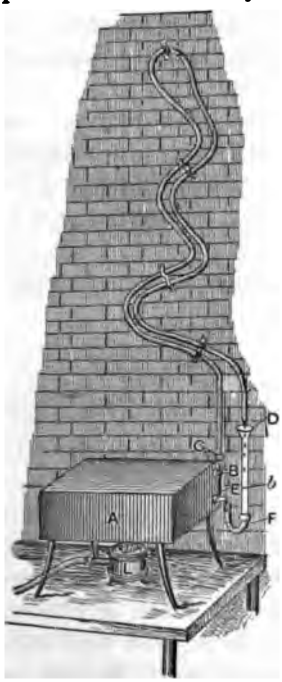
Water.	Albuminoid ammonia	Organic nitrogen as ammonia.
Brookline, Filter Gallery—		
June	0.0046	0.0190
July	0.0046	0.0220
August	0.0042	0.0170
Newton, Filter Gallery—		
June	0.0044	0.0190
July	0.0072	0.0160
August	0.0052	0.0100
Revere, Wells—		
June	0.0018	0.0034
July	0.0034	0.0070
August	0.0018	0.0100
Revere, Reservoir—		
June	0.0034	0.0130
July	0.0046	0.0150
August	0.0060	0.0190
Waltham, Filter Gallery—		
June	0.0030	0.0170
July	0.0050	0.0160
August	0.0056	0.0150
Waltham, Reservoir—		
June	0.0042	0.0230
July	0.0042	0.0180
August	0.0060	0.0160

It will be noted that the organic nitrogen in the surface waters is in general about double the albuminoid ammonia. The average of all the analyses of these waters given above is 0.0214 for the albuminoid ammonia, and 0.0476 for the organic nitrogen. In the ground waters the relation of the albuminoid ammonia to the organic nitrogen is still less. Too much importance must not, however, be given to this relation in the case of the ground waters, for it is probable that the figures given for the organic nitrogen are all a little high, owing to the fact that all of the sources of error were not fully known when most of the analyses were made. The proportional excess in case of the surface waters is believed to be insignificant, but with the smaller content of nitrogen in the ground waters the error is perhaps proportionately large.

A CONSTANT WATER BATH.

BY DR. BENNETT F. DAVENPORT.

A description of this apparatus will be found in the Pharmaceutische Rundschau for March, 1887, and in the Proceedings of the American Pharmaceutical Association for 1887. It has proven so satisfactory that I think it worth while to repeat the description here for the information of chemists. I have kept my bath boiling day and night seven days in the week, and have added water to it but 5 times in as many years.



It consists of the square box A, supported over a Fletcher's solid-flame burner. The top of the box, 15x15.5 inches, is formed by a brass plate, ⅛ inch thick, which thus is stiff enough to support a considerable weight without yielding, the sides and bottom being sheet-copper. From the point B projects a ½ inch brass tube, BC, which turns up at a right angle. At E is a stop-cock, which is connected by a thick rubber tube with the glass tube DF, which is fastened against the adjoining wall. Connected with C by a rubber-joint, is a ½ inch block-tin tube of 20 feet length, which extends up the wall in the manner shown to the highest point T, and thence returns and ends just over the slightly funnel-shaped top of the glass tube at D. The bath being filled with water to just the level Bb, may be kept constant by boiling for many days without appreciable loss of water, the steam being condensed in its passage up, or if uncondensed before it reaches the point T, in its passage down the block-tin tube. In flat-bottom platinum or porcelain capsules evaporation goes on very rapidly when placed on top of this water-bath. The whole surface of the bath is nickel plated.

THE DETECTION OF IODINE.

BY W. H. SEAMON.

The reaction of potassium iodide with the platinic salts, mentioned in Watt's Dict. of Chem. Vol. III. p. 288, furnishes a valuable test for the soluble iodides, being characteristic and delicate. The manner of making the test is as follows:

To the solution in a test tube add one or two drops of solution of platinic chloride. As the platinic chloride mixes with the liquid a beautiful red coloration is produced due to formation of platinic iodide. If much iodide be present the solution becomes black with subsidence of a brownish precipitate.

Free potassa, ammonia and hydrochloric acid require the addition of a large amount of the platinic chloride. It is therefore best to have it neutral or acid with sulphuric acid. The reaction is plainly visible if $\frac{1}{13000}$ pt. of iodine be present and is readily seen, though faint, with $\frac{1}{65789}$ pt. of iodine.

The test has been subjected to a large number of trials to prove its value with most satisfactory results.

SCHOOL OF MINES, ROLLA, MO., July 1, 1889.

THE SANITARY EXAMINATION OF WATER.

The following circular letter and list of questions in regard to the sanitary examination of water explain themselves. They have been sent by the Chairman of the Committee to such chemists as are supposed to be specially interested in this subject. At his suggestion we print them in this number of the Journal in the hope that they will thus be brought to the notice of others, whose names have not occurred to the Committee, who may like to take part in the discussion. From any such he will be glad to receive replies to the questions, before August 10th, 1889, or as much earlier as possible.

DEAR SIR:

At the last meeting of the American Association for the Advancement of Science the Committee of Section C, on methods of stating the results of water analysis was continued, and it was directed, further, to ascertain and report what can be accomplished towards securing on the part of water analysts uniformity in the methods of examining water used for drinking, and especially as to the organic matter.

It was the decided opinion of the meeting at which this action was taken, that a very great advantage would be gained, in respect to the sanitary study of waters, if at least a certain part of the chemical examination should be conducted on a common plan.

In consideration of the great and increasing importance of this sanitary question, and

the more and more prominent position that the chemist is coming to occupy in the consideration of the question, and of the very evident fact that these methods of examination for organic matter etc., are often not based on sharp and well defined reactions, making it altogether likely, if indeed not almost inevitable that different results should be obtained with different management of the details of the operations concerned; considering also the evident desirability that the confidence of the public in the judgment of the chemists should not be weakened by differences among the chemists themselves, we trust that you will be disposed to join in this movement by coöperating with this committee, and to give such time to the matter now as may be necessary for the answering of the accompanying questions, so far as your own practice leads you to take a practical interest in the subject.

Yours very truly,

G. C. CALDWELL, Ithaca, N. Y.
J. W. LANGLEY,
W. P. MASON,
J. A. MEYERS,
W. H. SEAMAN,
R. B. WARDER,
Committee.

THE COMBUSTION METHOD.

1. Do you use the Frankland combustion process, and if so, does your experience lead you to believe that its general adoption would lead to more useful and concordant results than are obtained by other methods in common use?

THE WANKLYN METHOD.

2. Assuming that you use this method, do you follow it precisely as described in the latest edition of Wanklyn's work on water analysis (1889)?

3. Referring to Mallet's paper in the Annual Report of the U. S. Board of Health, 1882; and assuming it to be allowed that the process as decribed by Wanklyn may be improved:

Would you not favor the adoption by general consent of the following suggestions made by him on page 208, based as they are on the results of his very extended investigations:

a. "In order to avoid uncertain ending of the collection of ammonia, whether free or albuminoid "stop the distillation" when and not before, the last measure of distillate contains less than a certain proportion—say 1%,—of the whole quantity already collected. This would in many cases involve the necessity of replenishing the contents of the retort with ammonia-free water."

b. "In order to diminish the loss of amines or other volatile forms of nitrogenous matter, a separate distillation should be made with alkaline permanganate added *at once*, in addition to

the usual course of treatment prescribed by Wanklyn—distillation begun with Na_2CO_3, and continued after the addition of $KMnO_4$; the results of the two distillations should be compared."

c. "In reporting the results obtained by the albuminoid ammonia process including the determination of free ammonia, the details of the evolution of ammonia, as collected by separate measures of distillate, should always be given."

3. Or in the place of *a* would you favor the adoption of Prof. Breneman's proposal (Jour. Am. Chem. Soc. **8**, p. 227) to collect a uniform number of portions of 10 cc. each, supposing that you follow the 100 cc. modification, 4 portions for the free ammonia, and 7 for the albuminoid?

4. Would you favor for general adoption the recommendations of Prof. Breneman (loc. cit.) of a uniform rate of distillation, namely 10 cc. per 10 minutes?

5. In the preparation of your permanganate solution do you follow Wanklyn or Mallet, loc. cit. p. 279?

6. Do you follow the 500 or the 100 cc. plan as given by Wanklyn?

7. Will you kindly describe and give reasons for any modifications of the "ammonia" process as described by Wanklyn that you have introduced in your own practice, and that you consider of sufficient importance for general adoption.

OXYGEN CONSUMED.

8. Do you follow Wanklyn, Tidy or Kuebel?

9. Assuming that, of course, you would prefer for general adoption the method that you follow: would you kindly give for incorporation in our report a brief statement of the reasons for your preference, and for important objections to the other methods?

10. Please describe and give reasons for any modifications of the process followed, that you have introduced in your own practice, that appear to you to be of sufficient importance for general adoption.

11. Do you regard the estimation of oxidized N. compounds as of sufficient importance for general adoption?

12. If yes, do you estimate them separately or together?

13. Will you please name the methods employed for these estimations, with reference to the place where they are described, and describe such modifications of the methods thus described as you have introduced in your own practice, and which appear to be of sufficient importance to be recommended for general adoption.

BIOLOGICAL EXAMINATION.

14. Do you make any use of biological examination of waters? Does your experience lead you to regard it as practicable and important for general adoption?

A NEW VOLUMETRIC METHOD FOR THE ESTIMATION OF FAT IN MILK, SKIMMED MILK, BUTTER-MILK, AND CREAM.*

BY CHARLES L. PARSONS.

There have been, within the last few years, so many improvements in breeds of cattle and in the production of milk, butter, and cheese, that no creamery-man, dairyman, or farmer can ignore them and still hold his own against the increasing competition of the day. Breeds of cattle and individual cows must be kept which pay for the food they consume by their milk, or they must be turned into beef. Butter must be made which comes up to the standard in flavor, color, and grain. Milk must be sold which meets the requirements of the law, where it is to be consumed as such, and where it is to be used in making butter, quality as well as quantity will soon determine the value. But, one may ask, on what basis shall one cow be kept and another rejected, or milk be bought and sold? The question has been many times answered. Quantity was formerly the basis, but now the amount of butter-fat which the milk contains must have equal consideration. The quantity is easily determined by measures or scales, but to determine the fat has been quite another matter.

Every butter-maker, in order to compete with others, must soon know the amount of fat in the original milk, the amount he has obtained in his cream, and the amount lost in the skimmed

*From advance sheets of the N. H. Agricultural Experiment Station, Appendix C. of First Annual Report.

milk and buttermilk. There has long been an increasing demand for some quick, simple, cheap, and accurate method which is applicable to all four. Buttermakers have been satisfied with general deductions from analyses at some distant laboratory, but now, from the demonstrated differences of the effect of individuals, breed, food, and temperature upon the churnability of cream, they must be made often and at home.

Different herds vary greatly in the quality of milk which they produce. This is shown plainly by the results of analyses given in the second annual report of the New York Dairy Commission. The average milk of seventy dairies there reported varied between 3.08 per cent. and 6.60 per cent. of butter-fat. It is manifestly unfair to every creamery and progressive farmer that the same price should be paid for milk containing three per cent. of fat as for milk containing double that quantity.

Many methods have been devised to be used for "testing milk." Feser's lactoscope, the lactobutyrometer, the creamometer, the oil-test churn, Heeren's milk-tester, the methods of Vogel, Soxhlet, and Short, and the lactocrite have all been proposed for general use and have met with varied success. Of these, the methods of Soxhlet and Short, and the lactocrite are undoubtedly the best. The expense of the lactocrite, and the ether required by the method of Soxhlet, however, shut them out from many creameries and dairies. Short's method requires no great expense, but while it gives good results on whole milk, in the words of its author it does not give "accurate results with milk containing under .50 per cent. of fat." This, of course, shuts out the analysis of most skimmed milk and some butter-milks. If a butter-maker is to balance accounts, the fat lost in the skimmed milk and butter milk must be taken into consideration, as well as the fat in the milk and cream.

I have for some time past been experimenting for the purpose of obtaining a simple and cheap method, applicable to all grades of milk and cream, which would give good results in the hands of any dairyman. The following is the method obtained, and at the request of the director of this station no scientific terms have been used where they could possibly be avoided :

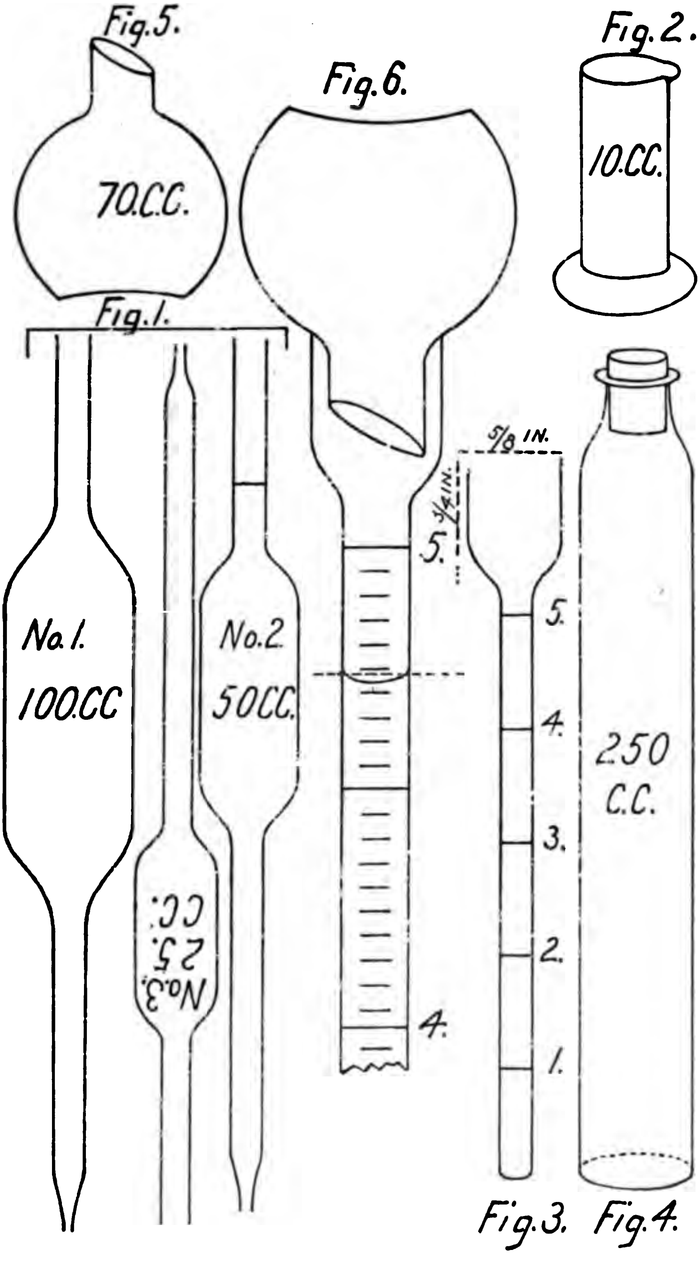


Fig. 5.
70.C.C.
Fig. 6.
Fig. 2.
10.CC.
Fig. 1.
No. 1.
100.CC
No. 3.
25.
CC.
No. 2
50CC.
5.
4.
5/8 IN.
3/4 IN.
5.
4.
3.
2.
1.
250
C.C.
Fig. 3. Fig. 4.

THE APPARATUS.

1. (*a*) Three pipettes (Fig. 1). No. 1, to hold 100 cubic centimeters; No. 2, to hold 50 cubic centimeters; No. 3, to hold 25 cubic centimeters, and having the lower stem at least nine inches long. (*b*) A measuring-cylinder (Fig. 2) holding, when full, 10 cubic centimeters. (*c*) A measuring-tube (Fig. 3) nine inches long and of such an internal diameter that the distance on the scale from zero to five shall be between seven and one-half and eight inches. These tubes are accurately calibrated, and are marked on the outside. The numbers 1, 2, 3, 4, and 5 on the scale represent so many whole cubic centimeters. The distance between 0 and 1, 1 and 2, etc., is divided by twenty lines, the distance between two of these subdivisions representing .05 of a cubic centimeter.

2. One or more long, slender bottles (Fig. 4), made of transparent glass, eleven inches high and one and one half inches in diameter, which hold about 250 cubic centimeters. The neck should fit a No. 2 rubber cork.

3. One or more flasks (Fig. 5) of about 70 cubic centimeters capacity, with the neck cut off obliquely, and of a size and length to go into the top of the measuring-tube, as in Fig. 6.

4. A drying-oven, eight inches wide, twelve deep, and fourteen high, which has a thermometer, shelf for holding flasks and measuring-tube, and a single-burner kerosene stove beneath for heating.

SOLUTIONS REQUIRED.

1. Gasoline, practically free from anything in solution. This gasoline is that ordinarily used in gas machines. It is sold in three grades (86°, 88°, and 90°), either of which will do. It begins to boil at about 80° F. and generally the last portions boil off at 140° F. (The substance used in street lamps generally called gasoline is not such, but naphtha, and will not do.)

2. Caustic soda solution. This is made by dissolving in an iron vessel one pound of commercial caustic soda in two pounds of water.

3. Strong alcohol (95 per cent), in which one ounce of castile soap to the gallon has been dissolved. To dissolve easily the soap should be scraped fine.

4. A little strong acetic acid.

DIRECTIONS FOR ANALYSIS.

Fill the largest pipette to the mark with the well-mixed milk and run into one of the bottles. Add to it the measuring-cylinder full of caustic soda solution and half full of alcohol soap solution. Then add pipette No. 2 full to the mark of gasoline. Cork the bottle. Shake hard for a few seconds, and again five or six times, at about equal intervals, in the next half hour. After the last shaking let the bottles stand a few minutes to see if the gasoline will not rise to the top. If it does not, add the measuring-cylinder half full of alcohol solution again, and as soon as bubbles cease to rise turn the bottles slowly upside-down and back again. This may be repeated until four portions of alcohol have been added, or separation takes place sufficiently to draw out the required quantity of the upper liquid. The bottles should be turned slowly in order not to mix again any of the gasoline solution which has separated. This separation generally takes place after the first addition of the alcohol solution.

As soon as the upper solution is perfectly clear, fill the smallest pipette to the mark with it, taking great care that none of the lower solution enters. From the pipette let the solution run into one of the flasks and evaporate the gasoline. Add two drops of acetic acid to the fat which is left behind. Place the flask in the oven and dry it at 245° to 255° F. for one hour and a half. Now turn the fat slowly into one of the measuring-tubes previously placed in the oven. Let the flask drip for ten minutes while the oven is at the same temperature. Remove the last drop by drawing the neck of the flask across the neck of the tube. Cool the measuring-tube until the first appearance of the fat solidifying, hold clasped in the hand a moment, and then read off the number on the scale which corresponds with the upper surface of the fat, as in Fig. 6. From the accompanying table ascertain the per cent. of fat which corresponds to this number.

Several analyses can be carried on at once, all flasks, bottles, and tubes being carefully numbered.

SKIMMED MILK AND BUTTER-MILK.

Skimmed milk and butter-milk are treated nearly the same as whole milk. The fat from either, if less than one per cent., need

be dried only three-quarters of an hour. If more than one per cent. it should be treated as a whole milk. It is easy after analyzing one or two milks to judge approximately from the amount of dried fat in the small flasks whether it is above or below this per cent. If the milk is thought to be below one per cent it should be measured in a tube which is already at least half full of dried fat. The reading on the scale for the fat already in the tube should be taken, and the reading after the addition of the fat from the flask. The difference between the two gives, of course, the reading for the skimmed milk or butter-milk.

CREAM.

The sample should be carefully mixed and free from air bubbles. Fill the largest pipette to the mark and let it run out into some suitable vessel. After blowing out the last drops, fill the same pipette with water and mix this well with the cream already measured. With this mixture fill pipette No. 2 to the mark, run it into one of the bottles, and proceed as for whole milk. The fat from very rich cream should be dried for two hours. It is always best to carry on two analyses of milk or cream, whatever method is used, the one to check the other. Results cannot be expected to agree as closely on cream as on milk, for the amount used is only one fourth as great ; nor is the accuracy, necessary in the one case, required for practical work in the other.

DETAILS.

Sampling.—The milk or cream should be well mixed and free from air bubbles. Fill the pipette a little above the mark on the neck by suction with the mouth, and quickly place a finger (dry) over the top. Then, by gradually letting in air, allow the milk to run out slowly until it just reaches the mark. All solutions measured with a pipette should be measured in this manner. These measurements should be made accurately, especially the measurement of the gasoline solutions. Always allow the pipettes to drip a moment, and blow out the last drops. In analyzing milk in creameries where the milk is tested only at intervals, the sample should be as nearly as possible an average one. To this end, mix the night's and morning's milk.

Treatment of the Gasoline Solution.—Do not try to hurry the

separation, as too much alcohol soap solution is worse than too little. The separation of whole milk or cream generally takes place within ten minutes. Skimmed milk and butter-milk occasionally require a longer time, but will always separate. Sour butter-milk does not separate as easily as fresh, and sometimes has to stand over night. If in filling pipette No. 3 any of the lower solution enters, the whole must be run back into the bottle and allowed to settle again. This need never occur if care is taken to draw up the solution slowly and not by jerks. Before using the small flasks for the first time, each one should have a little pure fat put in and melted. This should be allowed to drip out under the same conditions as in actual analysis. (Butter which has been melted and the salt and curd allowed to settle out, or the fat obtained in analysis, is best for this purpose.) A film of fat is thus left behind, which compensates for the fat left behind in analysis. This film is always approximately the same, as in evaporating the gasoline it is each time washed down by the gasoline condensing on the sides of the flask, and is again left when the fat is turned into the measuring-tube.

Evaporating Gasoline.—Gasoline takes fire very easily, and should not be evaporated within several feet of a flame, unless protected. It boils much below the boiling point of water. The best way for general use is to heat a pan or pail of water to boiling, and then carry it some distance from the fire. Now take the little flask containing the gasoline and fat in the hand and hold it on the hot water. The gasoline will immediately begin to boil, and in a short time will mostly pass off into the air, leaving the fat and a little gasoline behind. Care must be taken that the gasoline does not boil over, for in that case some of the fat would be lost. If it shows any tendency to do this, lift the flask from the hot water a moment, and the boiling will stop. The water should not be allowed to cool too much before evaporating The boiling gasoline will not burn the hand, as it boils at too low a temperature. The acetic acid is added to prevent the fat becoming gelatinous, as it frequently does without it. In creameries where steam is always to be had, a large number may be evaporated at once. Bore some holes in the top of a small closed box ; these should be about the size of the bottom of the flasks. Place the

flasks containing the gasoline and fat on top of these holes, and pass steam very slowly into the box through a rubber pipe. This is by far the easiest way, and requires only about five minutes for any number desired. The flow of steam must be under control, so that it may be stopped at any moment should the gasoline boil so fast as to be in danger of boiling over. Only a very little steam is required.

Drying the Fat.—The flasks containing the fat are placed on the shelf in the oven. The thermometer bulb should be on a level with the flasks. The oven is kept between 245 and 255 degrees Fahrenheit, and the flasks are dried at this temperature for the full time. If for more than five minutes the temperature should fall below 245, the flasks should be allowed to stay enough longer to make up for time thus lost. The flasks should drip into the measuring-tubes full ten minutes, with the temperature at the required height.

Reading the Measuring-Tubes.—After the fat is ready to measure, the tube is cooled down in the air, or, by taking care that none enters the tube, with water. As soon as the fat begins to grow opaque through solidification, the part of the tube containing fat is held clasped in the hand a moment until it clears up, when the reading is taken, which corresponds with the upper slightly curved surface of the fat, as is represented by the dotted line in Fig. 6. The reading in this figure is 4.74. Considering 1 on the scale as 100, 2 as 200, 3 as 300, etc., each sub-division equals five. This is perhaps the best way to read the scale, as decimals are avoided.

Cleaning the Apparatus.—Always rinse out pipette No. 1 with water, and pipette No. 3 with gasoline, when through using them. To clean pipettes, thoroughly fill them with some of the caustic soda solution, taking care that none of it gets in the mouth, and let them stand some time. Then turn it back and rinse well with water. Pipettes Nos. 2 and 3 should be dried before again using for gasoline. The flasks should be cleaned in the same way whenever necessary. It must be remembered, however, that a film of fat is intended to remain on the inside, and as long as nothing but fat is present cleaning is unnecessary. The tubes are cleaned carefully after each analysis by warming them, turning

out the fat, and rinsing each one twice with gasoline while still warm. Dry before using.

Using the Tables.—Find the first two figures of the reading obtained from the scale in the left hand column headed cc. Then pass horizontally towards the right to the column which has the third figure of the reading for its heading, and the per cent. of fat corresponding to the reading of the scale is found.

RESULTS.

In order to carefully test the above method, I have made a large number of analyses of the milk of individual cows, and a smaller number of analyses of skimmed milk, buttermilk, and cream. In every case these analyses have been duplicated by gravimetric methods, or the method of Soxhlet, either in my hands or by Mr. E. H. Farrington. To test it further, printed instructions were given to Mr. A. H. Wood, superintendent of dairy department of Station, to Mr. H. L. Barnard, station clerk, and to Mr. Fred D. Fuller, a student in the Agricultural College, none of whom were accustomed to chemical work. Each made several analyses with good success, as will be seen from the following results:

Milk Analyzed.	Number.	Reading of scale on tubes.	Breed.*	New method. Analyses by C. L. Parsons.	Gravimetric. Analyses by C. L. Parsons.
				Per Cent.	Per Cent.
Whole milk.	1	2.90	J.	5.81	5.84
"	2	1.62	D.	3.07	3.00
"	3	2.73	J.	5.43	5.58
"	4	1.90	D.	3.64	3.77
"	5	3.40	J.	6.97	6.91
"	6	1.92	D.	3.68	3.72
"	7	1.40	D.	2.63	2.71
"	8	2.67	J.	5.30	5.39
"	9	1.82	D.	3.48	3.56
"	10	3.10	J.	6.27	6.40
"	11	1.82	D.	3.48	3.46
"	12	3.05	J.	6.15	6.11
"	13	1.93	D.	3.70	3.70
"	14	2.85	J.	5.70	5.64
"	15	2.04	D.	3.93	3.95
"	16	1.70	D.	3.23	3.25
"	17	1.94	D.	3.72	3.70
"	18	3.15	J.	6.38	6.42

*J., Jersey; D., Durham; A., Ayrshire; H., Holstein.

Milk Analyzed	Number	Reading of [illegible]	Bureau*	New method [illegible]	[illegible]
				Per cent	Per cent
Whole milk	19	[illegible]	J	[illegible]	[illegible]
"	[illegible]	[illegible]	D	[illegible]	[illegible]
"	21	[illegible]	D	[illegible]	[illegible]
"	22	[illegible]	D	[illegible]	[illegible]
"	[illegible]	[illegible]	D	[illegible]	[illegible]
"	[illegible]	[illegible]	D	[illegible]	[illegible]
"	[illegible]	[illegible]	D	[illegible]	[illegible]
"	[illegible]	[illegible]	D	[illegible]	[illegible]
"	[illegible]	[illegible]	I	[illegible]	[illegible]
"	[illegible]	[illegible]	D	[illegible]	[illegible]
"	[illegible]	[illegible]	I	[illegible]	[illegible]
"	[illegible]	[illegible]	D	[illegible]	[illegible]
"	[illegible]	[illegible]	D	[illegible]	[illegible]
"	[illegible]	[illegible]	J	[illegible]	[illegible]
"	[illegible]	[illegible]	A	[illegible]	[illegible]
"	[illegible]	[illegible]	D	[illegible]	[illegible]
"	[illegible]	[illegible]	H	[illegible]	[illegible]
"	[illegible]	[illegible]	H	[illegible]	[illegible]
"	[illegible]	[illegible]	D	[illegible]	[illegible]
"	[illegible]	[illegible]	J	[illegible]	[illegible]
"	[illegible]	[illegible]	I	[illegible]	[illegible]
"	[illegible]	[illegible]	D	[illegible]	[illegible]
Cream	[illegible]	[illegible]		[illegible]	[illegible]
	[illegible]	[illegible]		[illegible]	[illegible]
	[illegible]	[illegible]		[illegible]	[illegible]
Same Cream	[illegible]	[illegible]		[illegible]	[illegible]
	[illegible]	[illegible]		[illegible]	[illegible]
	[illegible]	[illegible]		[illegible]	[illegible]
	[illegible]	[illegible]		[illegible]	[illegible]
Cream	[illegible]	[illegible]		[illegible]	[illegible]
Same Cream	[illegible]	[illegible]		[illegible]	[illegible]
	[illegible]	[illegible]		[illegible]	[illegible]
	[illegible]	[illegible]		[illegible]	[illegible]
	[illegible]	[illegible]		[illegible]	[illegible]
Cream	[illegible]	[illegible]		[illegible]	[illegible]
	[illegible]	[illegible]		[illegible]	[illegible]
	[illegible]	[illegible]		[illegible]	[illegible]
Skimmed milk	[illegible]			[illegible]	[illegible]
	[illegible]	[illegible]		[illegible]	[illegible]
		[illegible]		[illegible]	
	[illegible]	[illegible]			
	[illegible]	[illegible]		[illegible]	[illegible]
Butter milk	[illegible]	[illegible]		[illegible]	
	[illegible]	[illegible]		[illegible]	[illegible]

* [illegible]

Milk Analyzed.	Number.	Reading of scale on tubes.	Breed.*	New method. Analyses by C. L. Parsons.	Soxhlet's method Analyses by E. H. Farrington.
				Per Cent.	Per Cent.
Whole milk	55	1.95	D.	3.75	3.63
"	56	1.95	A.	3.75	3.73
"	57	2.27	A.	4.42	4.42
"	58	2.44	J.	4.79	4.82
"	59	1.83	D.	3.50	3.63
"	60	1.96	A.	3.77	3.93
"	61	2.05	A.	3.96	4.03
"	62	2.20	A.	4.27	4.39
"	63	2.05	D.	3.96	4.11
"	64	2.05	A.	3.96	4.11
"	65	2.53	A.	4.99	5.07
"	66	2.38	A.	4.66	4.63
"	67	2.50	J.	4.92	4.71
"	68	2.30	J.	4.48	4.42
Buttermilk	69	0.64	...	1.19	1.17
"	70	0.97	...	1.79	1.72
"	71	1.40	D.	2.63	2.76
"	72	0.70	D.	1.28	1.28
"	73	0.40	J.	.72	.81
"	74	0.87	...	1.60	1.64
"	75	0.64	...	1.17	1.33
"	76	0.82	...	1.50	1.61
"	77	0.64	...	1.17	1.30
Skimmed milk	78	0.24	...	.44	.32
"	79	0.30	...	.54	.44
"	80	0.18	...	.33	.22
"	81	0.22	...	.40	.37
"	82	0.29	...	.53	.55
"	83	0.30	...	.54	.52
"	84	0.17	...	.31	.23
"	85	0.31	...	.55	.50
"	86	0.17	...	.31	.30

Milk Analyzed.	Number.	Reading of scale on tubes.	Breed.*	New method. Analyses by C. L. Parsons.	Gravimetric. Analyses by E. H. Farrington.
				Per Cent.	Per Cent.
Whole milk	87	2.05	D.	3.96	3.89
"	88	2.02	D.	3.89	3.79
"	89	3.06	J.	6.17	6.30
"	90	1.85	D.	3.54	3.70
"	91	1.77	D.	3.38	3.53
"	92	2.88	J.	5.77	5.88

*J., Jersey; D., Durham; A., Ayrshire; H., Holstein.

Milk Analyzed	Number	Reading of scale in tubes	Breed *	New method Analyses by [illegible]	Lactocrite Analyses by [illegible]
				Per cent	Per cent
Whole milk	93	3.02	J	6.[illegible]	[illegible]
"	94	2.92	D	5.56	[illegible]
"	94	2.91	D	5.[illegible]	[illegible]
"	95	1.53	D	2.[illegible]	[illegible]
"	96	2.15	D	4.17	[illegible]
"	97	1.93	A	3.[illegible]	[illegible]
"	98	2.00	A	3.56	[illegible]
"	99	2.27	A	4.42	[illegible]
"	100	2.08	J	5.[illegible]	[illegible]
"		1.84		3.52	[illegible]
Skimmed milk		0.10		.18	[illegible]
"		0.12		.22	[illegible]
"		0.13		.24	[illegible]
"		0.13		.24	[illegible]
"		0.26		.17	[illegible]
		0.26		.44	[illegible]

Milk Analyzed	Number	Reading of scale in tubes	Breed *	New method Analyses by [illegible]	Lactocrite Analyses by [illegible]
				Per cent	Per cent
Whole milk	[illegible]	[illegible]	D	[illegible]	[illegible]
	[illegible]	[illegible]	D	[illegible]	[illegible]
	[illegible]	[illegible]		[illegible]	[illegible]
	[illegible]	[illegible]		[illegible]	[illegible]
Cream	[illegible]	[illegible]		[illegible]	[illegible]
	[illegible]	[illegible]		[illegible]	[illegible]

Milk Analyzed	Number	Reading of scale in tubes	Breed	New method Analyses by [illegible]	Lactocrite Analyses by [illegible]
				Per cent	Per cent
Whole milk	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]		[illegible]	

* [illegible]

In order to give the method a further test, and an eminently practical one, Mr. T. A. Hazen of the Norwich, Vt., creamery, consented to try it. Written instructions were delivered to him, together with ten bottles, ten flasks, six measuring-tubes, and the rest of the apparatus as given. His creamery superintendent, Mr. Smith, made thirty analyses; Mr. Hazen himself made eight. These eight were on the mixed night's and morning's milk of herds whose milk Mr. Smith had analyzed the day before, and although the analyses were made by two different men, on two succeeding days, the greatest variation between their results was six one-hundredths of one per cent. It was not possible to make gravimetric analyses for comparison, but Mr. Hazen reports that they found no trouble whatever in carrying out the method, and that he was much pleased with the results. He further stated that he thought with the apparatus furnished to him he could make about forty analyses a day.

Cost of Apparatus, etc.—Apparatus consisting of six bottles, six flasks, three measuring-tubes, three pipettes, a measuring cylinder, oven, thermometer, and kerosene stove can be bought for about ten dollars. The cost of the solutions used is small, being about one cent for an analysis. As it may sometimes be well to have the oven needed made at the nearest tinsmith's, the following directions for its construction are given: The oven should have five sides made of tin. The bottom should be one solid piece of sheet iron. The top should have three holes, one near the middle three quarters of an inch in diameter, in which a cork is placed for holding the thermometer, and two smaller holes to admit of air passing in and out. The oven should have a shelf, full size of the oven, nine and a half inches from the bottom. The shelf has a number of holes cut in it for holding the measuring-tubes. One of the wider sides should have a door six inches high and twelve wide, which occupies the upper six inches of that side. A Florence, Monitor, or Baby American single-burner kerosene stove is excellent for the purpose of heating. With a little practice, no trouble will be found in getting and keeping the necessary temperature. The dimensions which I would recommend for the oven are those already given in the description of "apparatus."

TABLE

FOR MILK, SKIMMED MILK, AND BUTTERMILK, CONTAINING FAT FROM .18 PER CENT TO 8.56 PER CENT.

cc.	0	1	2	3	4	5	6	7	8	9
0.1	.18	.20	.22	.24	.26	.27	.29	.31	.33	.35
0.2	.36	.38	.40	.42	.43	.45	.47	.49	.51	.53
0.3	.54	.56	.58	.60	.62	.63	.65	.67	.69	.71
0.4	.72	.74	.76	.78	.80	.81	.83	.85	.87	.89
0.5	.90	.92	.94	.96	.98	1.00	1.02	1.04	1.06	1.08
0.6	1.09	1.11	1.13	1.15	1.17	1.18	1.20	1.22	1.24	1.26
0.7	1.28	1.30	1.32	1.34	1.36	1.37	1.39	1.41	1.43	1.45
0.8	1.46	1.48	1.50	1.52	1.54	1.56	1.58	1.60	1.62	1.64
0.9	1.65	1.67	1.69	1.71	1.73	1.75	1.77	1.79	1.81	1.83
1.0	1.85	1.87	1.89	1.91	1.93	1.94	1.96	1.98	2.00	2.02
1.1	2.04	2.06	2.08	2.10	2.12	2.13	2.15	2.17	2.19	2.21
1.2	2.23	2.25	2.27	2.29	2.31	2.33	2.35	2.37	2.39	2.41
1.3	2.43	2.45	2.47	2.49	2.51	2.53	2.55	2.57	2.59	2.61
1.4	2.63	2.65	2.67	2.69	2.71	2.73	2.75	2.77	2.79	2.81
1.5	2.83	2.85	2.87	2.89	2.91	2.93	2.95	2.97	2.99	3.01
1.6	3.03	3.05	3.07	3.09	3.11	3.13	3.15	3.17	3.19	3.21
1.7	3.23	3.25	3.27	3.29	3.31	3.33	3.36	3.38	3.40	3.42
1.8	3.44	3.46	3.48	3.50	3.52	3.54	3.56	3.58	3.60	3.62
1.9	3.64	3.66	3.68	3.70	3.72	3.75	3.77	3.79	3.81	3.83
2.0	3.85	3.87	3.89	3.91	3.93	3.96	3.98	4.00	4.02	4.04
2.1	4.06	4.08	4.10	4.12	4.14	4.17	4.19	4.21	4.23	4.25
2.2	4.27	4.29	4.31	4.33	4.35	4.38	4.40	4.42	4.44	4.46
2.3	4.48	4.50	4.52	4.55	4.57	4.59	4.61	4.64	4.66	4.68
2.4	4.70	4.72	4.74	4.77	4.79	4.81	4.83	4.86	4.88	4.90
2.5	4.92	4.94	4.96	4.99	5.01	5.03	5.05	5.08	5.10	5.12
2.6	5.14	5.16	5.18	5.21	5.23	5.25	5.27	5.30	5.32	5.34
2.7	5.36	5.39	5.41	5.43	5.45	5.47	5.49	5.52	5.54	5.56
2.8	5.59	5.61	5.63	5.66	5.68	5.70	5.72	5.74	5.77	5.79
2.9	5.81	5.83	5.85	5.88	5.90	5.92	5.94	5.97	5.99	6.01
3.0	6.04	6.06	6.08	6.11	6.13	6.15	6.17	6.20	6.22	6.24
3.1	6.27	6.29	6.31	6.34	6.36	6.38	6.40	6.43	6.45	6.47
3.2	6.50	6.52	6.54	6.57	6.59	6.62	6.64	6.66	6.68	6.70
3.3	6.73	6.75	6.77	6.80	6.82	6.85	6.87	6.89	6.92	6.94
3.4	6.97	6.99	7.01	7.04	7.07	7.09	7.12	7.14	7.16	7.18
3.5	7.20	7.22	7.25	7.28	7.31	7.33	7.35	7.38	7.41	7.43
3.6	7.45	7.47	7.49	7.52	7.54	7.57	7.59	7.61	7.64	7.66
3.7	7.69	7.71	7.74	7.76	7.79	7.81	7.83	7.86	7.88	7.91
3.8	7.94	7.96	7.99	8.01	8.03	8.06	8.08	8.11	8.14	8.17
3.9	8.19	8.22	8.25	8.27	8.29	8.31	8.34	8.36	8.38	8.41
4.0	8.43	8.46	8.48	8.51	8.53	8.56				

CREAM, 15 PER CENT TO 45.60 PER CENT.

cc.	0	1	2	3	4	5	6	7	8	9
1.9	15.01	15.09	15.18	15.27	15.35	15.43	15.52	15.60	15.69	15.77
2.0	15.86	15.94	16.03	16.11	16.20	16.29	16.37	16.46	16.55	16.64
2.1	16.73	16.81	16.90	16.99	17.08	17.16	17.25	17.34	17.43	17.52
2.2	17.60	17.69	17.78	17.87	17.95	18.04	18.13	18.22	18.31	18.40
2.3	18.48	18.57	18.66	18.75	18.84	18.93	19.01	19.10	19.19	19.29
2.4	19.37	19.46	19.55	19.64	19.73	19.82	19.91	20.00	20.09	20.17
2.5	20.26	20.35	20.44	20.54	20.63	20.72	20.81	20.90	20.99	21.08
2.6	21.17	21.26	21.36	21.45	21.54	21.63	21.72	21.81	21.90	22.00
2.7	22.09	22.18	22.27	22.36	22.45	22.54	22.64	22.73	22.82	22.92
2.8	23.01	23.10	23.20	23.29	23.38	23.47	23.56	23.66	23.75	23.85
2.9	23.94	24.03	24.13	24.22	24.31	24.41	24.50	24.60	24.69	24.79
3.0	24.88	24.98	25.07	25.17	25.26	25.35	25.45	25.54	25.64	25.73
3.1	25.83	25.92	26.01	26.10	26.20	26.29	26.39	26.49	26.59	26.68
3.2	26.77	26.87	26.97	27.07	27.17	27.26	27.36	27.46	27.55	27.65
3.3	27.74	27.84	27.94	28.04	28.13	28.23	28.33	28.42	28.52	28.62
3.4	28.72	28.81	28.91	29.00	29.10	29.20	29.30	29.40	29.50	29.60
3.5	29.70	29.80	29.90	30.00	30.10	30.19	30.29	30.39	30.49	30.59
3.6	30.69	30.79	30.89	30.99	31.09	31.19	31.29	31.39	31.49	31.59
3.7	31.69	31.79	31.89	31.99	32.10	32.20	32.30	32.40	32.50	32.60
3.8	32.71	32.81	32.91	33.01	33.11	33.21	33.31	33.41	33.51	33.61
3.9	33.72	33.82	33.93	34.03	34.13	34.24	34.35	34.45	34.55	34.65
4.0	34.75	34.85	34.96	35.06	35.16	35.27	35.38	35.48	35.59	35.69
4.1	35.80	35.91	36.01	36.11	36.21	36.31	36.41	36.52	36.62	36.73
4.2	36.83	36.94	37.04	37.15	37.25	37.36	37.47	37.57	37.68	37.78
4.3	37.89	38.00	38.10	38.21	38.31	38.42	38.53	38.64	38.74	38.85
4.4	38.96	39.07	39.18	39.28	39.39	39.50	39.61	39.71	39.82	39.93
4.5	40.04	40.15	40.26	40.37	40.48	40.59	40.70	40.81	40.92	41.03
4.6	41.14	41.25	41.36	41.47	41.58	41.69	41.80	41.91	42.02	42.13
4.7	42.24	42.35	42.46	42.57	42.68	42.79	42.90	43.01	43.12	43.23
4.8	43.34	43.45	43.57	43.68	43.79	43.90	44.02	44.13	44.25	44.36
4.9	44.47	44.59	44.70	44.82	44.93	45.04	45.15	45.27	45.38	45.59
5.0	45.60									

THE INFLUENCE OF SILICON ON THE DETERMINATION OF PHOSPHORUS IN IRON.*

BY THOMAS M. DROWN, PROFESSOR OF ANALYTICAL CHEMISTRY IN THE MASSACHUSETTS INSTITUTE OF TECHNOLOGY, BOSTON.

The process for determining phosphorus in iron now in most general use in the laboratories of iron and steel works, is, I think, the one proposed by Mr. Emmerton.† In this process the solution of the iron for precipitation by ammonium molybdate is effected by treating the iron with nitric acid, evaporating to hard dryness in a dish, taking up with hydrochloric acid and driving off the greater portion of the hydrochloric by nitric acid. This procedure accomplishes, first, the destruction of all the carbonaceous matters; and, second, renders all the silica insoluble.

Experience has proved the former—the complete oxidation of the carbon—to be absolutely essential; but the explanation usually given, namely, that unless the carbon compounds are destroyed they will have a solvent action on the yellow precipitate, I am inclined to doubt. It seems to me more probable, as suggested by Mr. P. W. Shimer, that the conditions which are favorable for the oxidation of the carbon compounds ensure the complete oxidation of the phosphorus to ortho-phosphoric acid, and that the reason of the imperfect precipitation of the phosphorus, when the solution of the iron in nitric acid is directly precipitated by molybdate solution, is that the phosphorus is not completely oxidized.

There have been many means proposed for destroying the carbon compounds—chromic acid, potassium chlorate, potassium permanganate, hydrogen peroxide and others—which accomplish the purpose without the necessity of evaporating the solution to dryness. The advantage of the latter method, however, has been supposed to be that in this way the silica was at the same time rendered insoluble.

The statement of Klein‡ that, in the determination of phosphoric acid in basic slag, it is not necessary to separate the silica, since the ammonium silico-molybdate is soluble in 3 per cent. nitric acid, led me to try the effect of precipitating the phosphorus in a solution of pig-iron without previously evaporating to dryness. The results agreed perfectly with others obtained in

*A paper read before the Amer. Inst. Min. Eng. †Trans., Amer. Inst. Min. Eng. XV, 92; this Journal 1, 93.

‡Chem. Zeitung, 10, 721.

the usual way after the removal of the silica ; and further investigation showed that the silicon, at least in the form in which it exists when pig-iron is dissolved in nitric acid, is not precipitated by the molybdate solution.

There is a very marked difference in the effect of nitric acid of different strengths on pig-iron, as regards the condition of the silicon. When the acid is strong (1.2 to 1.4 sp. gr.) much of the silicon becomes gelatinous and prevents filtration, while with more dilute acid the silicon goes almost entirely into solution.

In a former communication to the Institute* I gave the effect of different strengths of hydrochloric acid on the silicon in pig-iron, showing that the strongest acid renders nearly all the silicon insoluble, and that weak acid dissolves it almost entirely. Generally speaking, the same is true, also, of nitric acid, as the following determinations show. It is interesting to note in the following figures that a nitric acid of medium strength dissolves more silicon than acid either stronger or weaker.

The pig-iron used in the following tests was a No. 1 foundry-iron, containing 2.42 per cent. silicon.

Effect of Nitric Acid of Different Strengths on the Silicon in Pig-Iron.

Acid.	Silicon, Per cent.	
Sp. Gr.	In Graphitic Residue.	In Solution.
1.4	2.28†	0.14
	2.08†	0.34
1.2	0.75†	1.67
	0.44†	1.98
1.16	0.07	. . . .
	0.07	. . . .
1.135	0.07	. . . .
	0.06	. . . .
	0.06	. . . .
	0.06	
1.116	0.09	. . . .
	0.11	. . . .

Acid.	Silicon, Per cent.	
Sp. Gr.	In Graphitic Residue.	In Solution.
1.102	0.12	. . . .
	0.12	. . . .
	0.12	. . . .
	0.12	. . . .
	0.13	. . . .
1.082	0.23	. . . .
	0.23	. . . .
	0.23	. . . .
1.070	0.20	2.12
	0.24	. . . .
	0.27	2.14
	0.31	2.12

*Trans. viii, p. 514.

†By difference.

The solutions obtained with the use of acid of 1.4 and 1.2 sp. gr. could not be filtered in concentrated condition, owing to the gelatinous form of the silica. They were, therefore, largely diluted and an aliquot portion of the solution was obtained by filtration. In this the silicon was determined.

The solutions obtained with acid of 1.116 sp. gr., and still more dilute, can be filtered without difficulty.

The separation of gelatinous silica is not wholly a matter of strength of acid, neither is it wholly a matter of the percentage of silicon in the pig-iron. It has often been noted that different pig-irons, under identical treatment with nitric acid, will filter at different rates; and sometimes the iron with least silicon proves most refractory in filtering. This suggests that silicon may exist in different conditions in pig-iron, a subject which cannot be further discussed in this paper.

Another pig-iron was tested in this series of experiments—a gray-forge iron, containing 0.63 per cent. of silicon—with the following results:

Silicon in Graphitic Residue.

	Per cent.
Acid, sp. gr. 1.2	0.19 0.20
Acid, sp. gr. 1.135	0.03 0.03 0.05 0.06

From the foregoing it will be seen that the strength of nitric acid most favorable for the solution of pig-iron is about 1.135 sp. gr., and not 1.2, which is generally used.* In the solution thus obtained there is no tendency of the silica to separate; not until the solution has been concentrated by boiling to small bulk does the silica begin to gelatinize.

When molybdate solution is added to a solution of pig-iron obtained in this way, the phosphorus, as might be expected, is but partially precipitated, because this treatment with nitric acid is insufficient to oxidize all the carbonaceous matters; or, as I prefer to believe, insufficient to oxidize all the phosphorus to ortho-phosphoric acid. To effect this object any of the oxidizing

*Acid of 1.135 sp. gr. is conveniently made by diluting acid of 1.2 sp. gr. with half its bulk of water, or by diluting 1.4 sp. gr., with water in the proportion of 3 to 7.

agents already mentioned may be used. Potassium permanganate, as proposed by Shimer,* seems to me the most convenient; but for the solution of the separated manganese peroxide I use, instead of hydrochloric acid a small quantity of some organic substance, such as tartaric, citric, or oxalic acid or sugar. The manganese oxide disappears almost immediately on the addition of the organic matter. After this treatment, which requires only a minute or two, the molybdate solution will give a complete precipitation of the phosphorus.

The following determinations were made in order to discover whether there is any tendency for the silicon in solution to be carried down with yellow precipitate, either mechanically or as silico-molybdate. The pig-iron used was that already mentioned, containing 2.42 per cent. silicon.

Silicon determined	I Per ct.	II Per ct.	III Per ct.	IV Per ct.
In residue from nitric acid solution,	0.040	0.040	0.070	0.100
In yellow precipitate,	0.030	0.050	0.012	0.012
In washings of the yellow precipitate with 2 per cent. nitric acid,	0.050	0.030	0.007	0.004
In filtrate from the yellow precipitate,	2.250	2.210	2.340	2.280
Total,	2.370	2.330	2.429	2.396

There seems to be, then, no evidence of the formation of any precipitate of ammonium silico-molybdate.

If the separation of the silica is omitted, the determination of phosphorus in pig-iron may be carried out in very much less time than is ordinarily required. In the laboratory of the Massachusetts Institute of Technology we have adopted the following procedure: About 1.5 grammes of the pig-iron (or more if very low in phosphorus) is treated in a covered beaker with 60 cc. of nitric acid of 1.135 sp. gr. (If preferred, 20 cc. of water may be first added, and then 40 cc. of nitric acid, sp. gr. 1.2. In this variation the action is at first a little less violent.) The beaker is heated on an iron plate until action ceases; three minutes is usually sufficient time if the borings are reasonably fine. The solution is then filtered from the graphite into a 500 cc. flask, and to the boiling filtrate is added 15 cc. of permanganate solution of 5 grammes to the liter.† Boiling is continued until the pink color has disap-

*Trans., xvii; this Journal **2**, 283.

†The strength of this solution is not a matter of importance; I have found the above amount sufficient. Shimer uses 10 cc. of a solution of 20 grammes to the liter.

peared, and a few very small fragments of tartaric acid are then added, causing the precipitated manganese peroxide to go almost immediately into solution. The amount of tartaric acid need not exceed 0.1 gramme, although the addition of an excessive quantity—as much as 1 gramme—does not influence the results perceptibly. To the clear solution 10 cc. of strong ammonia (sp. gr. 0.90) are added to partially neutralize the free nitric acid. This amount leaves the solution still amber-colored; it should not be so far neutralized as to become wine-colored. When the solution has cooled down to 90° C., 80 cc. of molybdate solution* is added, and the flask is closed with a rubber stopper, wrapped in a towel and shaken vigorously for five minutes, as recommended by Emmerton. From this point on the directions of Emmerton, in his paper already mentioned, are followed, namely, reduction by zinc and titration by permanganate. If fine, powdered zinc is used it may be entirely dissolved in about ten minutes, but filtration from the undissolved zinc seems generally to be preferred.

The following results were obtained in this way on the sample of pig-iron containing 2.42 per cent of silicon, in comparison with the older method of dissolving the iron in nitric acid, evaporating to dryness to separate silica and precipitations by molydate solution and magnesia mixture.

No. 1 Pig-Iron Containing 2.42 per cent. Silicon.
Phosphorus Determinations.

By the Old Method.	By the New Method.	By the New Method (Continued.)	
Per cent.	Per cent.	Per Cent.	Per cent.
0.284	0.272	0.297	0.286
	0.279	0.296	0.294
	0.290	0.289	0.294
	0.280	0.291	0.288
	0.291	0.291	0.292
	0.292	0.278	0.288
	0.289	0.294	
	0.292	0.293	

The rate of oxidation of the reduced molybdenum sesquioxide, on exposure to the air, is very slow, and is not ordinarily taken into account. Mr. J. W. Loveland found that it amounted, in this series of experiments, to 0.001 per cent. of phosphorus for

*Made by dissolving 100 grammes of molybdic acid in 400 cc. ammonia, sp. gr. 0.96, and adding 1000 cc. nitric acid, sp. gr. 1.2.

every ten minutes standing, before titration with permanganate. All the foregoing results have been corrected accordingly.

If rapidity of determination is of the first importance, a few minutes may be saved by omitting the filtering off of the graphite. Many trials show that this omission does not affect the accuracy of the results, although the removal of a foreign insoluble substance from the liquid enables one to carry out the process with greater satisfaction.

The following determinations of the phosphorus in the sample of gray-forge iron containing 0.63 per cent. silicon, were made without filtering off the graphite, by Mr. Greeley, a student at the Massachusetts Institute of Technology.

Gray-Forge Iron Containing 0.63 Per cent. Silicon.
Phosphorus Determinations.

By the Old Method.	By the New Method, without filtering from graphite.	
Per cent.	Per cent.	Per cent.
0.632	0.620	0.627
0.635	0.625	0.629
	0.631	0.645
	0.640	0.635
	0.633	

The determinations made in the solution of the same pig-iron, after filtering from graphite, gave 0.636 and 0.631. Further results obtained by Mr. Greeley are given below:

Bessemer Iron Containing 1.18 Per cent. Silicon.
Phosphorus Determinations.

By the Old Method.	By the New Method.
Per cent.	Per cent.
0.084	0.083
0.087	0.085
	0.086
	0.086
	0.085

No. 1 Pig-Iron Containing 2.29 Per cent. Silicon.
Phosphorus Determinations.

By the Old Method.	By the New Method.
Per cent.	Per cent.
0.334	0.324
0.330	0.327
	0.334
	0.324
	0.322

The process for steel is, of course, the same as for pig-iron, except that the question of filtration from graphite does not arise.

In a sample of Bessemer steel the following percentages of phosphorus were obtained.

By the Old Method. Per cent.	By the New Method. Per cent.
0.061	0.064
	0.060
	0.061

By one familar with the details of the process, a determination of phosphorus in pig-iron can be easily made in less than an hour.

I am indebted to Mr. James W. Loveland, Assistant in Chemical Analysis in the Institute of Technology, for valuable aid in this investigation; the credit for the analytical work, except as otherwise mentioned, is due entirely to him.

A NEW METHOD OF MILK ANALYSIS FOR THE USE OF DAIRYMEN, AND A COMPARISON OF ITS RESULTS WITH THOSE OBTAINED BY THE CHURN.*

BY G. H. FAILYER AND J. T. WILLARD.

For the use of dairymen, creameries, etc., it has seemed very desirable to have some simple way of testing the relative quality of milk. Such a method, to be practicable, would necessarily include these characters: It must be so simple, so far as manipulations are concerned, as to be easily performed by one who has not had training in a chemical laboratory. The appliances must be comparatively inexpensive. This would involve dispensing with a delicate balance, and hence would be a volumetric method, unless, by operating on a considerable quantity of milk, the heavier weights involved could be taken on a coarse balance. There have been various methods suggested for making this test. These have been of two classes: the one depending upon the principle of the churn, the other involving the determination of the total fat by some chemical or physical method. Of the latter class, perhaps the most successful is that known as Short's method. It is described in the Sixteenth Bulletin of the Wisconsin Experiment Station.† In all chemical methods, the total fat in the milk

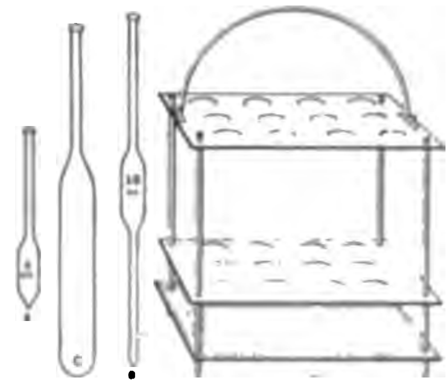
*From Report of the Kansas Experiment Station for 1888.

†This Journal **2**, 371.

is determined. Comparisons of milk made by the fat actually present will give the real food value, in this respect, of the milk as such. This has been the basis almost universally used in the past for judging the value of milk. Even the old method of testing by the lactometer, or by obtaining the specific gravity, had the fat in the milk in view. No account was taken of any difference in the proportion of this fat that could be obtained by churning. All methods which completely extract the fat from the milk are alike in this respect. So that none of these have advantages over the others except in respect to accuracy, and to ease and rapidity of performance. The Short method, to which reference has been made, is rather simple, so far as the manipulations are concerned, but the chemical reactions involved are somewhat complicated. The accuracy of the work, however, does not depend upon a knowledge of the principles involved in these reactions, if the very full directions given in the bulletin are carefully followed. But one drawback, which will greatly operate against the general use of the method, is the fact that, including measuring, cooling, and the four hours' boiling, fully five hours will be required to complete the analysis. Of course the operator can be doing other work during much of this time, but some personal supervision would seem to be necessary. Where many analyses are being performed at the same time, this element of time will not be of such consequence; but if only a few analyses are to be made, it seemed to be very desirable to have a shorter method than this. An attempt was made to work out such a method. It was decided at the outset that the butter should be determined as such. The problem seemed to be simply to dissolve the curd and collect the butter fat. After trying several solvents and combinations of these, a modification of a process described by Dr. W. Schmid in the *Zeitschrift für analytische Chemie*, Vol. 27, p. 464, was finally settled upon as being the best, all things considered.* The method, as originally proposed, consists in boiling ten cubic centimeters of milk with an equal quantity of strong hydrochloric acid in a graduated tube until the liquid turns brown. It is then cooled, and shaken up with thirty cubic centimeters of ether. After allowing it to settle, the volume of su-

*This Journal **2**, 439.

pernatant ether is observed, and ten cubic centimeters of this ether are taken off and evaporated in a tared dish. The dish and contained fat are accurately weighed, and from this and the known volume of ether from which the ten cubic centimeters were taken the total fat in the milk is calculated. This method requires an accurate balance, and some skill in using it. Where the necessary appliances are at hand, this may be entirely satisfactory. We have given it no trial in this respect, our aim being to avoid weighing. It was found that the solution of the curd could be readily effected in the hydrochloric acid, but that the fat did not readily collect as a whole. The use of a solvent for this purpose seemed essential. To make the readings volumetric, and at the same time delicate, a tube was used having a contracted part, similar to those described by Short, except that the bottom is thin and rounded and well annealed, so that the contents can be boiled over a naked flame, and the narrow tube, in which the reading is effected, is of less diameter so that smaller differences will be made apparent.



The details of the process as finally settled upon will be made clear below.

The following materials are necessary for this method of analysis:

a. Tubes in which the analysis is made. Ours were made from eight inch test tubes by drawing off the top and sealing on a piece of tubing of 4.5 mm. internal diameter, and expanded into a small funnel at the top. The whole tube is [illegible] inches long, the narrow portion being [illegible] inches. The narrow tube is graduated. (See Fig. C.)

b. A water bath of some sort, about [illegible] inches deep, and having a rack inside on which the tubes can be supported. A rectangular box of copper is the best, but a cheaper one may be used. A [illegible] pail would do, in which case the rack would better be

round. Fig. D illustrates a very serviceable form of rack for holding the tubes. It is 5x6½ inches, and 6½ inches high. The lower shelf is made of perforated metal, and the upper two have holes to accommodate twelve tubes.

(*c*) Some means of boiling the water in the bath. A gasoline stove would be best, where gas is not available.

(*d*) Two pipettes, as illustrated by A and B in the cut. It is better to have the 8 cc. pipette of the form shown, as it can then be put down in the bottle of acid and filled without suction, the acid fumes making the latter method very disagreeable. In using pipettes, the liquid to be measured is drawn up above the mark on the stem, and is then allowed to fall until the lowest portion of the surface of the liquid in the tube just reaches the line. That remaining will be the required volume. In measuring the milk, at least one-half minute must be allowed for the milk to drain down, the last drop being blown out. The pipette for milk must be kept clean. This is easily done by rinsing it repeatedly with cold water, *immediately* after using. Finish the cleaning by rinsing with hot water.

(*e*) A glass tube of about 4 mm. external diameter, 18 inches long, and bent at right angles about 5 inches from one end. A small rubber cork on the other end assists one in blowing, or in connecting with a bellows.

(*f*) Where a Bunsen gas burner cannot be used, an alcohol lamp giving a large flame.

(*g*) Something for introducing boiling water into the analytical tube. A wash bottle is best; a pipette may be used. A tin cup with a spout soldered in near the top would answer every purpose, and be unbreakable. The spout should be a tube, like that of an oiler, the opening being very small.

(*h*) A bellows, which can be attached by a rubber tube to the glass tube described in (*e*), is very desirable, but not essential, as the blowing can be done by the lungs. A small rubber bulb pump might be used.

(*i*) Concentrated commercial hydrochloric acid.

(*j*) A gasoline of low boiling point, such as used in gas

machines. This must leave no residue when evaporated by the method used in the analysis.

In making the analysis, 10 cc. of the milk are first carefully measured into the tube used in analysis. The inside of the narrow, graduated portion should be wet with water first. There will then be no difficulty in introducing the milk if the tube is held in an inclined position. The milk is then heated just to boiling by* a naked flame (gas is best, but a large alcohol lamp will answer) and 8 cc. of conc. hydrochloric acid added from a pipette, taking care to wash down any milk in the narrow part of the tube. The mixture is then heated cautiously in the flame, passing the tube rapidly backwards and forwards across the flame, holding it in an inclined position, and so that the heat is applied to the *upper* portion of the liquid. There is considerable tendency to foam at first. As soon as the foam is seen to be rising, the tube must be removed from the flame momentarily, and heated again as soon as the foam subsides. In about a minute all foaming ceases. If now the contents of the tube be examined, it will be seen that a flocculent layer rises rapidly to the surface. This contains the butter fat, entangled, however, with some portion of the other milk solids which is not readily soluble in acids.

The next object is to get rid of this substance entangling the fat. This portion of the operation is the only one requiring special care; and upon its proper execution depends the success of the analysis. The contents of the tube must be kept very hot; they must be kept constantly shaken together; the heating must be continued long enough and yet not too long. The tube is best shaken by holding it in a considerably inclined position and moving it rapidly backwards and forwards, in a way which gives it both a lateral and a longitudinal motion, thus setting the contents of the tube into rapid rotation. This method of giving the best motion, while hard to describe, is easy of execution. It is not necessary to shake in this particular way, but the contents must be kept thoroughly mixed. Care must be taken not to shake the contents against the opening of the narrow tube, or

*If the acid and milk are mixed cold, a coagulated mass is produced, similar to ordinary thick milk. This mixture is much less readily shaken and boiled, and solution less readily effected, than if treated in the manner described.

they will be thrown out by the rapidly escaping steam and acid. During heating, the flame must never be applied to the bottom of the tube, but to a point on the side which would be above the liquid if it were at rest, but is kept constantly covered by the vigorous shaking. By heating and shaking in this way, there is absolutely no danger from bumping. The heating is continued until, on stopping for a moment, the globules of fat are seen to be clear as they slide down the side of the tube, and in the layer which they form on the liquid. The acid liquid in the tube will have become black, and the flocculent matter which at first rose with the fat will have disappeared. If the heating is not continued long enough, this matter is not all dissolved, and will rise with the fat when the measurement is made, and prevent accurate reading. If, on the other hand, the heating be too long continued, the acid effects further decompositions, and a black, humus-like solid is produced, which interferes equally as much with the measurement of the fat. The heating requires about five minutes. When it is judged to be complete, the tube is set aside to cool somewhat, or is dipped in cold water a few minutes. When it is cooled below the boiling point of the gasoline, but while the butter fat is still melted, 15 or 20 cc. of gasoline are added. The contents of the tube are gently agitated, until the fat is mostly taken up; it is then shaken vigorously, the opening being kept tightly closed by the finger. If any of the frothy mixture produced lodges in the narrow part of the tube, it must be allowed to flow back before removing the finger, or the inside pressure will drive it out. The tube is rinsed down with a little gasoline and set aside. In a few minutes the gasoline containing the fat will have risen to the surface in a clear stratum.

The tube is now placed in the bath of boiling water, laid in a very much inclined position, so that the gasoline is spread over a large surface, and a current of air is blown in by means of the narrow glass tube, either by the mouth or by a bellows. The current of air is continued until the gasoline is all driven off. The tube is then filled with boiling water up to where it begins to contract, being careful not to stir the contents much. The tube is twirled a few times to detach any small globules of fat

which may have lodged on the side, and is then allowed to stand in the bath of boiling water for a few minutes to allow the fat to collect in one globule, and to be sure to expel all traces of gasoline. This is not only necessary to accurate measurement of the fat, but if any gasoline remains it is liable to volatilize suddenly and drive out the fat if it has been brought up into the narrow tube. Finally, enough more boiling water is added to bring the column of fat within the graduated tube, and its amount is measured. The measurement is best made by having the hot water bath nearly full, so that readings can be made while the body of the tube is immersed. In this way the column of fat remains stationary, while, if the tube is taken out, the cooling and consequent contraction of the water causes it to move rapidly downward, and makes accurate measurement very difficult. If a light-brown flaky substance rises in the water, lodging against the fat, or rising between it and the glass, it indicates that the milk was not boiled with the acid long enough. If, however, a black substance rises in the same way, the boiling was too long continued. Sometimes a twirl of the tube will send the interfering substance down long enough to enable the reading to be made. Sometimes a thin platinum wire is useful in displacing it. If the heating has been properly conducted, however, there will be no trouble from this source.

The analysis of a single sample of milk occupies about twenty-five minutes. Four can be analyzed in an hour and a quarter if the water has been put to heating, so as to be boiling when wanted.

It will occur to the chemist that ether might serve a better purpose in collecting the fat. It was, however, found entirely unavailable on account of its solubility in water. While ether can be readily removed from fat alone, it cannot be removed from a layer of fat floating on water containing ether in solution. The ether continually passes from the water to the fat until entirely removed from the water. This was found to require a long time. Sometimes, too, a sudden ebullition takes place, which is very likely to cause the loss of some of the fat. Gasoline, by its insolubility in water, presents no such difficulties.

The calculations necessary depend in part on the kind of graduation adopted for the tubes in which the analyses are made. This may be simply a millimeter scale on the narrow tube. In this case, it is necessary to know the capacity of the tube for a given length. It is better to have the tube graduated in cubic centimeters and fractions. It can easily be graduated in fiftieths of a cubic centimeter. If ten cubic centimeters of milk be taken for analysis, the number of tenths of a cubic centimeter of fat measured will give the per cent. by volume. If the specific gravity of the hot butter fat be taken as $\frac{87}{100}$, the per cent. by volume, multiplied by this fraction, will give the per cent. by weight, if we assume that the milk delivered by a 10 cc. pipette weighs ten grams. Perhaps a still better kind of graduation would consist in providing the tube with a decimal scale, such that 87 divisions shall be equal to one cubic centimeter. One-tenth the number of divisions occupied by the column of fat will show the per cent. by weight in the milk.

If it be assumed that a 10 cc. pipette will deliver ten grams of milk, no calculations other than those indicated above will be necessary. For ordinary analyses for purpose of comparison, this assumption will give results which are sufficiently near the truth. If greater accuracy is desired, a correction must be introduced. Milk being heavier than water, ten cubic centimeters will weigh more than ten grams. On the other hand, a 10 cc. pipette, allowed to drain one-half minute or even a minute, will not deliver ten cubic centimeters of milk; so that an accurate correction cannot be applied by taking into account the specific gravity of the milk.

The following trials were made, bearing on this point. An accurate 10 cc. pipette was used. The contents of the pipette were measured out a number of times in succession, and all weighed at once. The milk was allowed to drain out one-half minute, except in the first instance, when it was allowed to drain one minute. The last drop was blown out. The specific gravity of the milk was taken by an accurate flask.

KIND OF MILK.	Average amount obtained from 10 cc. pipette.	Weight of 10 cc., as calculated from the specific gravity.	Per cent. delivered.	Number of times pipette was filled.	
Night's milk stirred up in the morning . .	10.2082	10.3250	98.9	5	Pipette drained one minute.
New milk	10.1755	10.3167	98.6	8	Pipette drained one-half minute.
New milk from another source	10.1755	10.3231	98.5	9	Pipette drained one-half minute.
Skim-milk	10.1934	10.3560	98.4	9	Pipette drained one-half minute.

It will be seen that the weight obtained in the last three trials is 1.5 per cent. less than that calculated from the specific gravity. On the other hand, it is in excess of ten grams by about 1.8 per cent.

From this we see, that if the per cent. of fat obtained by the method of calculation before described be diminished by $\frac{1}{60}$ of itself, the result will be very close to the truth. There will probably be a slight variation with the specific gravity and other properties of the milk.

To test the accuracy of the method, the analyses given in the table were made. Proper tests of a method require that there should be parallel determinations in each of several distinct analyses, and that the results of these should be compared with the real amount of the substance known to be present. Where a definite and pure compound is operated upon, the proportion of any ingredient present is easily calculated. But in case of such a substance as milk, comparison of the results of a new method of analysis must be made with the results of an already tried and accepted one. In this case, comparisons were made with the method used by Dr. Babcock in his work at the New York Experiment Station. Duplicate analyses were made by this method except in one case. All analyses made are printed ; in some cases there were three, in some four, by the new method.

Table giving results of analyses of milk by the new method, and by Babcock's gravimetric method.

DATE.	NEW METHOD.						GRAVIMETRIC METHOD.			
	Parallel Analyses.				Average.	Greatest difference.	Duplicate Analyses.		Average.	Greatest difference.
Dec. 20...	4.52	4.54	4.42	. . .	4.49	.12	4.61	4.52	4.57	.09
Dec. 21...	5.20	5.33	5.34	5.37	5.31	.17	5.27	5.25	5.26	.02
Dec. 22...	4.91	4.81	4.76	4.88	4.84	.15	4.86	. . .	4.86	. .
Dec. 24...	4.95	4.90	5.02	. . .	4.96	.12	4.89	4.92	4.91	.03
Dec. 25...	4.56	4.50	4.53	. . .	4.53	.06	4.62	4.61	4.62	.01
Dec. 26*	4.53	4.35	4.57	. . .	4.48	.22	4.70	4.78	4.74	.08
Dec. 27*	4.84	5.03	5.27	. . .	5.05	.43	5.03	5.04	5.04	.01
Dec. 29...	3.63	3.51	3.56	. . .	3.57	.12	3.49	3.53	3.51	.04
Dec. 31*	5.02	4.91	5.31	. . .	5.08	.40	5.03	5.08	5.06	.05
Jan. 2.....	5.16	5.20	5.10	. . .	5.15	.10	5.25	5.32	5.29	.07

In the analyses of the milk of December 26 and December 27, the acid was left in contact with the milk much longer than usual. In those of the milk of December 31, the boiling was less prolonged than usual. The bad effect of excessive and of insufficient action of the acid are exemplified by the much more widely divergent results obtained on those days. Leaving out these results, a comparison of the figures will show that the greatest difference between the results of parallel analyses by the new method is .17 per cent., and the greatest departure of any single determination by the new method from the mean result obtained by the gravimetric analyses of the same milk is .19 per cent. Usually the difference is only .1 to .12 per cent. If averages are to be compared, the differences are much less. The results are certainly satisfactory for technical purposes.

It is not claimed for this method any greater accuracy than by

*Method modified, as stated in the text.

Short's method; from the manner of executing the work in the two cases, it seems that equal care should give almost equal accuracy. Both assume certain constants which are averages, and which will depart from the truth in individual cases, but for practical purposes will answer fairly well, especially if the milk is the mixed milk of a herd, for then it will come nearer being of average character. The short time necessary to complete the analysis, the simple reagents required and the fact that all the butter is collected are the principal advantages of the new method of analysis.

It was also part of the scheme of comparison to churn the milk in order to see how the results of analysis agree with those by the churn. A Cherry churn was used to make the butter test. Two-quart glass fruit jars were used on the churn. In these were placed one kilogram (about 2.2 pounds) of the fresh milk. This filled the jars about three-fifths full. In each trial three exactly similar portions were weighed out. For three successive churnings the milk was let stand twenty-four hours and then churned. It was perfectly sweet and the whole milk was churned, as it seemed a fairer test than to introduce the error of unequal skimming. In the last three trials, six portions of milk were weighed out as before. All were left 48 hours; three were kept cool and remained sweet; the other three were kept warm enough to become sour. The churning of all was effected at a temperature of 21 or 22 degrees C. (70 to 72 degrees F.), except the milk of December 26; this was churned at a higher temperature; as a result the butter was whiter than usual, and collected in balls so that it was worked to remove water collected in cavities in the balls and then weighed "moist." Otherwise this butter was treated as were the others described below. Except as noted above the butter procured was eminently granular and had a fine color. Visitors to the laboratory, who saw the butter as it was drying or after being bottled, spoke of its excellent appearance. There can be no question as to the satisfactory character of the method of churning. Any unsatisfactory results by the churn must be explained otherwise than by the manner of churning. The butter was placed on brass gauze and thoroughly washed with cold water. It was then allowed to drain, after which it

was weighed. This is the "moist" weight of the table. The butter was then allowed to dry in the air for twenty-four hours, being still in the granular condition and piled up much like shot. After twenty-four hours' drying, the butter was weighed, giving the "dry" weight of the table, and immediately bottled. The butter was afterwards analyzed for water and butter fat. The difference between these two per cents. and one hundred per cent. was considered "curd." The following tables give our results upon the six samples of milk. They show the per cent. of fat in the milk; the per cent. of butter obtained, weighed both moist and dry; the composition of the dry butter, and the per cent. of fat extracted as butter from the milk.

Table showing per cent. of fat in the milk, per cent. of butter obtained, the composition of the butter, and the per cent. of fat extracted as butter from the milk. Milk set 24 hours, and churned sweet.

DATE.	Per cent. of fat in milk	PER CENT. OF BUTTER.		COMPOSITION OF BUTTER.			Per cent. of fat extracted as butter from the milk.
		Weighed moist.	Weighed dry.	Per cent. of fat.	Per cent. of water.	Per cent. of curd by difference.	
Dec. 25	...	4.65	4.24	78.99	18.51	2.50	3.35
Dec. 25	...	5.10	4.58	74.40	23.21	2.39	3.41
Dec. 25	...	4.94	3.80	78.45	18.91	2.64	2.89
Mean	4.62	4.90	4.21	77.28	20.21	2.51	3.25
Greatest difference	...	.45	.78	4.59	4.70	.25	.43
Dec. 26	...	4.50	4.23	72.18	24.77	3.05	3.05
Dec. 26	...	5.03	4.77	71.36	25.34	3.30	3.40
Dec. 26	...	4.97	4.64	66.82	29.28	3.90	3.10
Mean	4.74	4.83	4.55	70.12	26.46	3.41	3.18
Greatest difference	...	.53	.54	5.36	4.51	.85	.35
Dec. 27	...	5.76	5.37	75.77	22.57	1.66	4.07
Dec. 27	...	5.89	5.59	78.33	20.45	1.22	4.38
Dec. 27	...	6.31	5.89	73.11	26.40	.49	4.30
Mean	5.04	5.99	5.62	75.74	23.14	1.12	4.25
Greatest difference	...	.55	.52	5.22	5.95	1.17	.31

Table showing per cent. of fat in the milk, the per cent. of butter obtained, the composition of the butter, and the per cent. of fat extracted from the milk as butter. Milk set forty-eight hours; one set of samples churned sweet, the other set churned sour.

DATE.	Per cent. of fat in milk.	CHURNED SWEET.						CHURNED SOUR.					
		Per cent. of butter obtained.		Composition of butter.				Per cent. of butter obtained.		Composition of butter.			
		Weighed moist.	Weighed dry.	Per cent. of fat.	Per cent. of water.	Per cent. of curd.	Per cent. of fat extracted as butter from the milk.	Weighed moist.	Weighed dry.	Per cent. of fat.	Per cent. of water.	Per cent. of curd.	Per cent. of fat extracted as butter from the milk.
December 29 . . .	. . . .	4.01	3.51	74.56	23.94	1.50	2.62	3.12	2.76	80.69	15.83	3.48	2.23
December 29 . . .	. . . .	4.23	3.78	72.39	25.26	2.35	2.43	3.09	2.90	72.95	25.98	1.07	2.11
December 29 . . .	. . . .	4.19	3.70	73.53	24.46	2.01	2.72	3.00	2.61	79.05	19.08	1.87	2.06
Mean	3.51	4.14	3.66	73.49	24.55	1.95	2.59	3.07	2.76	77.23	20.30	2.14	2.13
Greatest difference . . .		.22	.27	2.17	1.32	.85	.29	.12	.29	7.74	10.15	2.41	.17
December 31 . . .	. . . .	5.16	4.71	78.64	19.88	1.48	3.70	5.81	5.48	83.11	15.03	1.86	4.55
December 31 . . .	. . . .	4.92	4.55	76.79	20.98	2.23	3.49	6.02	5.64	81.21	17.04	1.75	4.58
December 31 . . .	. . . .	5.04	4.66	80.28	17.84	1.88	3.74	. . . .	. . .	. . . .	. . . .	. . .	. . .
Mean	5.06	5 04	4.64	78.57	19.57	1.86	3.64	5.92	5.56	82.16	16.04	1.81	4.56
Greatest difference . . .		.24	.16	3.49	3.14	.75	.25	.21	.16	1.90	2.01	.11	.03
January 2	. . .	5.18	4.81	77.34	20.05	2.61	3.72	6.65	6.21	76.50	20.92	2.58	4.75
January 2	. . .	5.51	5.06	76.90	21.18	1.92	3.89	7.29	6.82	70.19	26.83	2.98	4.79
January 2	. . .	4.69	4.32	79.57	18.02	2.41	3.44	7.60	7.05	67.54	29.36	3.10	4.76
Mean	5.29	5.13	4.73	77.94	19.75	2.31	3.68	7.18	6.69	71.41	25.70	2.89	4.77
Greatest difference . . .		.51	.74	2.67	3.16	.69	.45	.95	.84	8.96	8.44	.52	.04

An examination of the tables will show that the yield of butter varied with circumstances with the greatest irregularity. Even with duplicates that are treated exactly alike, the difference is sometimes about twenty per cent. of the least quantity. On December 25, the least is .78 of one per cent. less than the greatest. On January 2, the difference is .74 per cent.; the difference on the last date in the case of the sour milk is .84 per cent. These figures are based on the weight of the milk. When calculated on the butter itself, they become respectively 20.5 per cent., 17.1 per cent. and 13.5 per cent.

If the milk be not treated alike, then the yield seems to vary still more. Thus, on December 29, the mean of one set, the sweet milk, was 3.66; of the sour milk, 2.76; a difference of .9 of one per cent. of the milk. Calculated on the least of the above means, it is practically one-third. On December 31, the means are 4.64 and 5.56; difference .92—equal to one-fifth of the less. On January 2, the mean of the yields of sweet milk is 4.73; of the sour, 6.69—a difference of 1.96 per cent. This is 41.4 of the smaller quantity. These from comparing means. If we look for the greatest difference from the same milk regardless of treatment, we get for December 29, 1.17 per cent. in favor of sweet milk; on December 31, 1.09 per cent in favor of the sour milk; on January 2, 2.73 per cent. in favor of the sour milk. In the last case, the difference between the least butter obtained and the most is 63.2 per cent. of the least. When these differences are compared with the per cents. themselves, it is seen that they are very great. There are certainly grave sources of error in the use of the churn for testing milk. Unless very great care be taken to have all conditions alike, the method would seem to be untrustworthy. In these trials there were a sufficient number of duplicates to make nothing further in this line desired. But had more than six different kinds of milk been used, it would have added to the value of the test. But where the results show a great variation with a small number, it is probable that the variations would have been increased by a greater number. One would reject a method on fewer unsatisfactory trials than would induce him to accept it if the results are satisfactory.

When the percentage of fat in the milk is compared with the "dry" butter, that is, drained butter, obtained by the churn, it

is seen that only a very general agreement in quantity exists. Sometimes analysis gives the greater per cent. and sometimes the churn does. The milk of December 27th, with 5.04 per cent. of fat, churned sweet, gave 5.62 per cent. of butter; but the milk of the 31st, with 5.06 of fat, churned sweet, gave only 4.64 of butter; and of January 2, with 5.29 per cent., gave 4.73 of butter. In these cases, although there was more fat, there was less butter. We might be inclined to see in this the proof of certain views as to the relation of composition to churnability of milk. But if we look further, we will see that December 31 and January 2, churned sour, gave butter in excess of the fat, being respectively 5.56 and 6.69. It will be seen that the quantity of butter obtained is less or greater than the fat in the milk according to the conditions of churning. Even the fat extracted in the butter varies similarly.

If we inquire why the amount of butter should so vary, the answer in general terms will be, that not all the fat is ever extracted from milk by the churn; that what we get and weigh as butter is made up of true butter fat, curd and water. These are in not at all constant proportions. This fact is brought out well in the part of the table showing the composition of the butter.

If we look at the composition of the butter, we see some reasons for these variations in the amount of butter obtained. With the same amount of real butter, it is evident that the greater the proportion of water and curd the greater will be the apparent yield of butter. By referring to the portion of the above table which gives the fat extracted as butter from the milk, it will be seen that, although these figures come within narrower limits, they are far from concordant. When the treatment has been the same, the results are close, but between the sour and the sweet there are marked differences. Thus, between the 4.58 per cent. and the 3.49 per cent., of December 31, there is a difference of nearly one-third of the latter. The difference between the 4.79 and the 3.44, of January 2, is two-fifths of the latter. These results, all taken together, prove, so far as proof can be drawn from the number of trials, that it is exceedingly difficult to get the same yield of butter from duplicates. Except as stated, the

conditions attending these trials were more nearly alike than will usually be the case in dairy trials; indeed, it was the aim to treat the duplicates alike, and in no respect is it possible to specify wherein this was not true; but the results differ, and there must have been some unexplained cause for it. It is possible that the yield of butter may be made to serve as a test of the quality of milk, but it is evident that the treatment given both milk and butter is by no means an indifferent matter. It was not, however, the purpose of these trials to work up the conditions by which accuracy can be secured with the churn, but incidentally these results have been obtained, and these conclusions drawn.

MILK ANALYSIS.

BY DR. BENNETT F. DAVENPORT.

In the discussion of the relative merits of the different methods of milk analysis which was published in this month's number of *The Analyst*, the many advantages of a simple modification of Wanklyn's method does not, I think, receive its due consideration. This modification, which I have adopted, I have already published in my Annual Report as Milk Inspector for the City of Boston, Mass., 1885, and as analyst to the Massachusetts State Board of Health in their Annual Report for 1886, page 138. As being now the method generally followed by most of the official milk analysts in New England, a simple description of it may be of interest to others.

The 5 gms. of milk are weighed off in a large flat-bottom platinum capsule of full 2½ inches diameter on the bottom, and about 3 inches across the top. The ½ inch high side turns up from the bottom not with a sharp angle, but slight rounding, this being about the curve with which the milk runs up the rim of the capsule drawn by capillarity. The milk in drying down does not thus form any thicker deposit at the angle of the side than elsewhere upon the about 5. square inches of bottom surface of the capsule. This relatively large amount of surface, one square inch to each gm. of sample taken, causes each inch to be covered with only a little over a single grain of dried milk solids. The deposit is therefore so very thin as to be readily exhausted of its fat in its subsequent

treatment with boiling petroleum naphtha. When using such small dishes as were originally proposed by Wanklyn the residue would be so thick, that is about three times, as would naturally render any such rapid method of extraction as I employ impossible, as the English analysts have learned. Each capsule has its serial number engraved upon it, and they are made to weigh a little over 25 gms. each, that the bottoms may be stiff enough to remain perfectly flat, a matter of very great importance. They are also made to differ from each other only in the second and third places of decimals, so that only those weights have to be changed in many weighings, which is a matter of no little saving of labor where many score of weighings are to be made each day. A table of their weights is kept within the case of the scales. These capsules containing their samples are placed upon a constant-level closed-top water bath of the peculiar construction described on page 269 of this number of the JOURNAL OF ANALYTICAL CHEMISTRY. This will hold 25 capsules at a time. Here, being surrounded by an atmosphere not already nearly saturated with moisture, as would be the case if they were upon a water bath with openings on its top, they quickly evaporate to apparent dryness. They are then transferred for their final drying to a constant weight to one of Weisnegg's large porcelain-lined air baths, regulated to the constant temperature of 105° C. Here they are dried in about half an hour, when they are cooled in a desiccator and each weighed immediately upon being taken out to guard against their rapid gain in weight from exposure to the air. Replaced upon the closed-top water bath the capsule is filled from a wash bottle with petroleum naphtha of the quality of the benzine of the U. S. Pharmacopoeia, revision of 1880. This, unlike ether, will dissolve out neither milk sugar nor lactic acid from the dried milk-solid residue. Moreover, one gallon of it costs but as much as one pint of ether would, which is a matter of some little consequence to one like me who has had to use about 100 gallons of it in milk analyses during the past five years.

The naphtha in the capsule, after being allowed to boil down about one-half, is decanted off against a rod into a basin to guard against the remote possibility of some flakes of milk residue being

poured off with it. Replaced upon the bath, the capsule is refilled with naphtha. This boiling up and decanting off is repeated three to four times, when after the last one the outside of the capsule is washed off with naphtha played upon it from the wash bottle to prevent any residue of fat being left there. The capsule then finally replaced upon the bath to dry off the naphtha, is then cooled and weighed as before, the butter being rather determined from the loss of weight in the solids than by the weight of the evaporated washings.

The ash is made by ignition of the capsule over a one inch wide Bunsen lamp, which thus gives so wide a flame as not to require a heating to a high temperature at one point, and thus a possible loss by the volatilization of the potassium chloride. The milk sugar I determine by the use of a Soleil-Ventzke saccharimeter in the same manner as has now been several times described in the journals by Drs. Wiley, Vieth and others. The other ingredients which reduce copper in Fehling's solution, are not thus reckoned as sugar, by which the apparent amount of albuminoids present, which are obtained by difference, would be diminished.

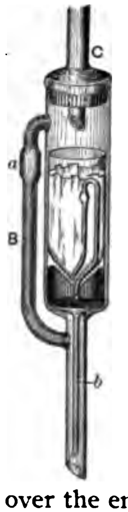
The peculiarities of my modification, is then the use of an evaporating capsule of such a very large relative area as will leave the residue thin enough to be readily exhausted by the boiling naphtha. This a diameter of 2½ inches in the capsule will do for 5 gms. of better than average quality of milk, with a surety that there will not be as much as one-tenth of one per cent. of fat left as a maximum error. And surely this is fine enough for all commercial work, and it is much to be doubted if much which affects to be more accurate by a decimal than this is really so. The method, as involving no transfer of substance, cannot well be made more simple for the determination of total solids, fat and ash. When the average life of such an evaporating capsule under reasonable usage is considered, it cannot well be cheapened, although the outlay for a kilo of platinum for 40 capsules like mine does, it is true, involve an original expense of nearly $400. The shape of my capsules makes possible completeness of extraction with the solvent used, and the construction of the closed water bath hastens the process in no small degree.

ABSTRACTS.

APPARATUS AND REAGENTS.

A Simple Air Bath.—T. O. Sloan (Sci. Am. **60,** 345) describes an air bath which for cheapness and simplicity is worthy of notice. It consists of an ordinary flower pot inverted over a sand bath or an iron plate or pan resting on a tipod. The hole in the bottom of the pot is used for the insertion of a thermometer, and if necessary for drying tubes, other holes may be drilled through the pot at any point. The top of the bath may be used for drying precipitates, the moisture in the paper being both absorbed and evaporated. S. C.

Apparatus for Fat Extraction.—R. Frühling, Ztschr. angew Chem. 1889, 242.



The apparatus consists of two parts—the outer part or holder closed at the top by a ground stopper through which passes the cooling tube *c.* and the inner part or receiver which carries the material to be treated.

The receiver is made slightly smaller than the interior diameter of the holder so that it may be easily removed and also that any ether that may condense on the walls of the holder can pass down to the heating bulb.

In treating coarsely pulverized material a tuft of glass wool or a layer of sand, previously ignited, is placed in the bottom of the receiver so that none of the material will be carried through the siphon. For finely ground substances a filter paper, folded and pressed over the end of a test-tube, is placed in the receiver.

By weighing the receiver, with the material in it, before and after extraction the weight of the residue may be obtained and when a filter paper is used the residue is left in convenient form for further analysis. J. E. W.

Determining the Strength of Liquids by Means of the Voltaic Balance.—G. Gore, Chem. News **59,** 243. The basis for this method is the fact that the greater the degree of concentration of a solution, the larger is the amount of dilution required to reduce its voltaic energy to a given magnitude. The author has previously described (Proc. Roy. Soc. **45,** 268) the method of measuring the voltaic energy of a substance, and has given the magnitudes of such energy induced in a voltaic couple of unamalgamated zinc and platinum by more than a hundred different substances in water solution, in comparison with the same couple excited by water alone, the temperature being the same in all cases. To determine the practicability of the voltaic balance method, aqueous solutions of nitric, hydrochloric and sulphuric acids, common salt, ammonia, and sodium carbonate were used, and the degree of strength of each was determined by chemical analysis, by specific gravity, and by the voltaic balance method. The proportions of water needed for one part by weight of the substance in order to reduce its voltaic energy to the same magnitude, viz., that required to just visibly deflect the needles of a galvanometer, were then ascertained, and these numbers also represent the relative magnitudes of voltaic energy of the substances used. A known volume of the substance was taken in each case, and the per cent. of substance determined by chemical analysis. The degree of strength of a second portion was determined by specific gravity. A third portion of known volume was then taken, its average amount of voltaic energy measured, and its degree of concentration found by the dilution necessary to reduce its energy to the same magnitude. The results given are:

	HCl	H_2SO_4	HNO_3	$NaCl$	Na_2CO_3	NH_3
Chem. Analysis . . .	1.85	5.60	2.97	9.13	7.21	1.05
Specific Gravity . . .	1.70	5.44	2.80	8.74	7.63	1.03
Voltaic balance . . .	1.65	5.70	2.90	8.71	7.57	1.06

Part of the differences are due to inequalities of temperature.

A. H. W.

New Determination of the Specific Gravity of Ammonia Solution.—G. Lunge and T. Wiernik, Ztschr. angew chem. 1889, 181.

Table of the Specific Gravity of Ammonium Hydroxide at 15° C.

(LUNGE AND WIERNIK.)

Sp. Gr. at 15°.	Per cent. NH_3	NH_3 contained in one Liter at 15°. Grammes.	Correction of the sp. gr. for ±1°.	Sp. gr. at 15°.	Per cent. NH_3.	NH_3 contained in one Liter at 15°. Grammes.	Correction of the sp. gr. for ±1°.
1.000	0.00	0.0	0.00018	0.940	15.63	146.9	0.00039
0.998	0.45	4.5	0.00018	0.938	16.22	152.1	0.00040
0.996	0.91	9.1	0.00019	0.936	16.82	157.4	0.00041
0.994	1.37	13.6	0.00019	0.934	17.42	162.7	0.00041
0.992	1.84	18.2	0.00020	0.932	18.03	168.1	0.00042
0.990	2.31	22.9	0.00020	0.930	18.64	173.4	0.00042
0.988	2.80	27.7	0.00021	0.928	19.25	178.6	0.00043
0.986	3.30	32.5	0.00021	0.926	19.87	184.2	0.00044
0.984	3.80	37.4	0.00022	0.924	20.49	189.3	0.00045
0.982	4.30	42.2	0.00022	0.922	21.12	194.7	0.00046
0.980	4.80	47.0	0.00023	0.920	21.75	200.1	0.00047
0.978	5.30	51.8	0.00023	0.918	22.39	205.6	0.00048
0.976	5.80	56.6	0.00024	0.916	23.03	210.9	0.00049
0.974	6.30	61.4	0.00024	0.914	23.68	216.3	0.00050
0.972	6.80	66.1	0.00025	0.912	24.33	221.9	0.00051
0.970	7.31	70.9	0.00025	0.910	24.99	227.4	0.00052
0.968	7.82	75.7	0.00026	0.908	25.65	232.9	0.00053
0.966	8.33	80.5	0.00026	0.906	26.31	238.3	0.00054
0.964	8.84	85.2	0.00027	0.904	26.98	243.9	0.00055
0.962	9.35	89.9	0.00028	0.902	27.65	249.4	0.00056
0.960	9.91	95.1	0.00029	0.900	28.33	255.0	0.00057
0.958	10.47	100.3	0.00030	0.898	29.01	260.5	0.00058
0.956	11.03	105.4	0.00031	0.896	29.69	266.0	0.00059
0.954	11.60	110.7	0.00032	0.894	30.37	271.5	0.00060
0.952	12.17	115.9	0.00033	0.892	31.05	277.0	0.00060
0.950	12.74	121.0	0.00034	0.890	31.75	282.6	0.00061
0.948	13.31	126.2	0.00035	0.888	32.50	288.6	0.00062
0.946	13.88	131.3	0.00036	0.886	33.25	294.6	0.00063
0.944	14.46	136.5	0.00037	0.884	34.10	301.4	0.00064
0.942	15.04	141.7	0.00038	0.882	34.95	308.3	0.00065

For temperatures other than 15° an example will illustrate the method of using the table.

Having found the sp. gr. at 13° to be 0.900, then $2 \times 0.00057 = 0.001$, the correction for 2°; $0.900 - 0.001 = 0.899$, the sp. gr. at 15°.

For temperatures higher than 15° the correction is to be added.

J. E. W.

On the Best Method of Preparing Nitrogen for Lecture Purposes.—Charles R. C. Tichborne, Chem. News **59**, 171.

10 gms. of ammonium sulphate and 10 of sodium nitrite are mixed in a retort of the capacity of half a liter or about 16 ounces, with 40 cc. of glycerine and 60 cc. of water. The retort is placed with the neck elevated to an angle of about forty degrees, so that the water may condense and fall back into the retort. A bent tube is fitted with a cork into the neck, and conducts the gas into a wash bottle, or it may be collected at once for general purposes without washing. 100 cc. of water may be used instead of the mixture of glycerine and water, but it does not seem to work quite so regularly. Heat is applied directly to the retort, and the disengagement of free nitrogen begins at a temperature a few degrees below the boiling point of water. It steadily but rapidly proceeds, the temperature generally rising a few degrees above the boiling point, but after a little time beginning to fall. A gentle but constant heat will, however, keep up the disengagement of gas until the nitrite is all decomposed. It is desirable to slightly increase the heat towards the end of the reaction. If the reaction is hurried too rapidly, ammonia is evolved. When carried on slowly, no ammonia, but a trace of nitrogen dioxide is produced, which may be easily removed by washing with dilute potassium permanganate solution. Almost pure and neutral nitrogen will be evolved by carefully regulating the temperature.

A. H. W.

Oxidations by Means of the Electric Current.—Edgar F. Smith, Chem. News **59**, 293. (From Journal of the Franklin Institute, April, 1889.)

Experiments were undertaken to oxidize the sulphur in chalcopyrite, as follows: 10 gms. of solid potassium hydroxide were placed in a small nickel crucible and carefully heated over a Bun-

sen burner until the water was expelled; then the flame was reduced so that its heat would just keep the contents of the crucible liquid. The mineral was then placed upon the alkali and the crucible was connected with the positive pole of a battery, while a heavy platinum wire, connecting with the negative pole, extended just under the surface of the fused mass. When the current was passed, a lively action ensued, but loss by spattering was prevented by placing a perforated watch crystal over the crucible. After ten minutes the connection was broken, the crucible was allowed to cool, placed in a beaker, covered with water, and then warmed on an iron plate. Hydrochloric acid was added, and the sulphur was determined as barium sulphate, which, when ignited, showed not the slightest trace of iron. The current used registered one ampere (10.45 cc. HO gas per minute) as it passed through the fused potassium hydroxide. About 10 gms. of potassium hydroxide were used for from .1 to .15 gm., but when working with from .2 to .3 gm. of mineral, the quantity of alkali should be doubled. The crucibles used were one and a quarter inches in height, and one and three-eighths inches wide, but the most convenient form would be that measuring two inches in length and one and a half inches in width. Thus far, the application of this method to the oxidation of the sulphur in pyrite or to chromite has not been successful, but with chalcopyrite very close results were obtained. A. H. W.

Testing the Purity of Cupric Oxide and Lead Chromate.—M. v. Nencki, Monatshefte f. Chem. **10**, 233. In an essay upon the "Testing of Chemical Reagents for Purity," Dr. Krauch, a chemist employed by E. Merck, gives the following method for testing copper oxide to be used in elementary analysis:

a. 100 gms. when heated in a current of air give off no acid vapors.

b. 2 gms. are dissolved in hydrochloric acid and diluted with water; the solution is almost clear. It is precipitated with sulphuretted hydrogen and the filtrate from the precipitate evaporated. This should leave only a small residue (a little iron).

c. The hydrochloric acid solution (1 : 50) is not rendered turbid either by barium chloride or sulphuric acid.

d. 20 gms. heated with 50 cc. water on the water bath, the water poured off and evaporated, leaves no weighable residue.

These tests according to Nencki are not sufficient, as he finds that most samples of copper oxide contain considerable amounts of lime, probably derived from calcareous water used for washing it. This is tested for by treating the copper oxide with warm, dilute acetic acid, precipitating any dissolved copper with sulphuretted hydrogen and adding ammonium oxalate to the filtrate to precipitate the lime. Some samples contained as much as 1.02 per cent., and only a few were free from it.

Lead chromate frequently contains lead oxide, which may be extracted by means of dilute acetic acid, in which the chromate is insoluble, and precipitated as sulphate.

INORGANIC ANALYSIS.

Estimation of Nitrogen in Nitrates by Ferrous Sulphate.—The apparatus consists of a graduated flask marked at 250 cc. provided with a rubber stopper pierced by two holes. The first carries a tube 5 to 6 cc. in length drawn out at its upper part; the second carries a tube which extends to within .5 cm. of the bottom of the flask. It is furnished at its upper end with a receptacle of 35 to 40 cc. capacity, of a cylindrical form and furnished with a glass stopcock. This apparatus serves not only to introduce the solution of nitrate which is to be analyzed, but also for the introduction at the commencement and end of the operation of a solution of bicarbonate of soda and carbonic acid which at the beginning drives the air out of the apparatus and at the end expels from the balloon the last traces of dioxide of nitrogen. For the titration of the monoxide of iron, a solution of bichromate of potash is employed, ferrocyanide of potassium being used in the case of the last reaction. The following solutions are necessary:

1. A solution of ferrous sulphate containing 100 gms. of ferrous sulphate and 75 cc. strong sulphuric acid per liter.

2. A solution of bichromate of potash containing 17.85 gms. pure and dry bichromate of potash per liter.

3. A standard solution of nitrate of soda containing 50 gms. of this salt, pure and dry, per liter.

4. A cold saturated solution of bicarbonate of soda.

METHOD OF OPERATION.

50 cc. of the ferrous sulphate solution is placed in the flask with about 25 cc. of strong sulphuric acid and well shaken. The flask is then closed with the stopper arranged as described. The flask is placed upon a large sand bath capable of holding five or six of like size. The stop-cock being closed, about 25 cc. of the solution of bicarbonate of soda is placed in the cylindrical funnel; the stop-cock is now operated so as to allow the bicarbonate of soda to flow into the flask drop by drop. This operation is continued until the funnel is empty, when 10 cc. of the solution of nitrate of soda is introduced which is allowed to flow in like manner into the flask in such a way that the evolution of the dioxide of nitrogen shall take place at about the same rate obtained by the carbonic acid preceding it. As the operation proceeds the contents of the balloon are seen to gradually turn brown. Before the funnel is completely emptied the stop-cock is closed, and when the brown color of the liquor has disappeared another portion of 25 cc. of the solution of bicarbonate of soda is introduced in the cylindrical funnel, which is allowed to flow in slowly as before. The flask is then taken from the sand bath, the cock having been closed. After cooling, the volume is made up to 250 cc. with distilled water, the flask well shaken and the remaining monoxide of iron titrated with a solution of bichromate of potassium. The number of cc. required gives by a simple calculation the percentage in nitrogen of the nitrate of soda. Some of the numbers obtained by this method are given as follows:

	Method of Schloesing-Grandeau.	Ferrous Sulphate.	Theory.
Nitrate of soda (pure)	16.45	16.45	16.47
Nitrate of soda (commercial) . . .	15.80	15.75	"
Another trial	15.40	15.45	"
"	15.65	15.60	"

(Bailhache, Comptes Rendus, Vol. 108, No. 21, page 1122.)

H. W. W.

The Proportion of Nitric Acid in the Rains of Tropical Regions.—The intensity and continuity of the electrical phenomena in the neighborhood of the equator led to observation upon the character of the rainfalls in equatorial regions.

One of the stations chosen was at Caracas, Venezuela, at 10°.3 and an altitude of 922 meters. The climate is characterized by a constant temperature, the annual variation being only 21°.8, by an unequal distribution of the rains and by the violence and frequency of storms. The observations extend over a period of two years and therefore give a reliable mean. Every rainfall during that period was collected and analyzed separately. There were 121 rain storms of which 63 occurred in the first year of observation and 58 in the second. The mean quantity of nitric acid per liter in the rain water was as follows:

From July, 1883, to July, 1884, ...	2.45 mgr.
From January, 1885, to December, 1885, . . .	2.01 mgr.
Mean for the two periods	2.23 mgr.

The richest in nitric acid was collected on the 19th of October, 1883, and showed 16.25 mgr. per liter. The poorest water showed only .20 mgr. per liter.

Boussingault found as the mean proportion of nitric acid in the station of Liebfrauenberg in Alsace, .18 mgr. nitric acid per liter. The mean number given by Lawes and Gilbert of Rothamsted, England, shows .42 mg. per liter.

There is therefore in the rains of equatorial regions much more nitrate than in those of temperate countries. The means obtained in Venezuela were more than 30 times greater than those of Boussingault, and 5½ times higher than those of Lawes and Gilbert.

This richness in nitric acid is attributed to the extreme electrical tension and frequent discharges of electricity which affect the oxidation of the nitrogen. In estimating the rain fall at that altitude at one meter, the quantity of nitrogen brought to the soil in the form of nitrate corresponds for each hectar of surface to 5,782 kilogrammes. Compare this with the amount found by Boussingault, viz: 330 kg., and by Lawes and Gilbert, viz: 830 kg., and the enormous excess of nitrogen in the rains of tropical regions is at once manifested.

In another tropical station situated at Saint-Denis, in Reunion,

at 20°.5 south latitude, Raimbault examined during the years 1886-87, 19 samples of rain water which showed a mean content of nitric acid per liter of 2.67 mg.

Estimating the rain fall at the same depth as above, viz., 1 meter the above figures show an addition of 6.93 kg. of nitric nitrogen to each hectare.

It is seen from the above that the quantity of nitrogen brought to the soil in our climate is negligible but it is not so in tropical regions where the rains furnish a true nitrogenous manure equivalent to about 50 gms. of nitrate of soda per hectare annually. There is no doubt but that this influx of nitrogen contributes to the luxuriant development of tropical vegetation. (Muntz & Marcono, Comptes Rendus Vol. 108, No. 20, p. 1062.)

H. W. W.

The Electrolytic Separation of Cadmium from Zinc.—Edgar F. Smith, and Lee K. Frankel, Am. Chem. J. **11**, 5, 352.—The authors find that using a cold solution of cadmium and zinc, with 4.5 g. KCN, the whole diluted to 200 cc. that in 18–23 hours all the cadmium will be precipitated without the zinc, using a current yielding 3 cc. oxygen and hydrogen per minute. The results are accurate.

C. W. M.

Quantitative Separation of Arsenic and Antimony.—O. Koehler (Chem. Zeit. Rep. **13**, 167), takes advantage of the solubility of sulphide of antimony in concentrated hydrochloric acid for the separation of these metals. He finds that arsenious acid may be completely precipitated from a hot concentrated hydrochloric acid (sp. gr. 1.124) solution by means of sulphuretted hydrogen while the antimony remains in solution. The precipitated sulphide is filtered off and washed with dilute hydrochloric acid until a drop of the filtrate gives no indication of antimony with sulphuretted hydrogen. The arsenic is determined by oxidizing the sulphide with bromine water and precipitating with magnesium mixture.

W. C.

Detection of Carbonic Oxide in the Air.—C. de LaHarpe and Frédéric Reverdin, Bull. soc. chim. [3] **3**, 163.

The air is filtered by passing through glass wool or cotton and passed over pure dry iodic acid, heated to 150° C. and then into a

solution of starch in distilled water. The carbonic oxide is changed to carbonic anhydride and a corresponding quantity of iodine set free which colors the starch blue. The iodic acid is placed in a small distilling flask and heated on an oil bath. Nine liters of air passed over the iodic acid in twenty minutes showed the presence of about .00001 to .00002 of carbonic oxide. C. W. M.

Modification of Bunsen's Apparatus for the Analysis of Manganese and Chromates.—C. de LaHarpe and Fréderic Reverdin, Bull. soc chim. LI, **3**, 164.

The modifications are slight and are as follows: In the upper side of the vessel in which the gas is absorbed are blown four cavities to hinder the rapid escape of the gas. Around the upper part of the delivery tube a large tube is placed and fitted by corks, forming a small condenser. This tube has two side tubes for supplying and carrying off water. C. W. M.

On the Volumetric Determination of Sulphates.*—H. Quantin, Bull. soc. chim. LI, **1**, 21.

The method described is as follows: 19. gms. of neutral potassium chromate and 50 to 100 cc. pure HCl are dissolved in water and the solution diluted to 200 cc., to this solution 24.35 gms. $BaCl_2$ in solution is added and the whole diluted to 1000 cc. and filtered. 50 cc. of this solution precipitate from 0.3 to 0.4 of sulphuric acid. It is necessary that the solution should contain an excess of potasium chromate with reference to the $Ba\ Cl_2$. If the $Ba\ Cl_2$ were in excess it would precipitate the H_2SO_4 without producing an equivalent quantity of chromic acid. The presence of an excess of chromate necessitates a correction which must be subtracted in every case. The correction is determined on a quantity equal to that used in actual analysis, by precipitation with ammonia. A portion of the filtrate is taken and to it a solution of double sulphate of iron and ammonium added until complete reduction is effected. The correction will be from 8.2–0.5 cc. It may be determined once for all by precipitating 100 cc. in a liter flask with an excess of ammonia and reducing the filtrate by the ferrous solution with addition of 5 cc. pure sulphuric acid. The ferrous solution is prepared by dissolv-

*See this Journal, **1**, 72.

ing 20 gms. of double sulphate of iron and ammonium and 10 cc pure sulphuric acid in water and diluting to one liter. For the analysis dissolve 1 gm. potassium sulphate in 60 cc., add 100 cc of barium chromate solution and agitate. When the liquid begins to clear, add ammonia in excess, producing a sulphur yellow color. Filter, and to 100 cc. of the filtrate add 5 cc. concentrated sulphuric acid. The iron solution is now run in from a burette, changing the solution to red, olive brown, green, and finally greenish blue. When the bluish color appears, drops of the solution are added to drops of a very dilute solution of ferricyanide of potassium on a porcelain surface, and the moment a blue color appears in a drop on the porcelain the titration is ended. The reaction is of great delicacy, the error never being more than one drop. The determination does not require more than half an hour at the most. The analysis is performed in the same manner. C. W. M.

On the Presence of Tin in Certain Kinds of Sugar, a Means of Detecting it and the Probable Influence of this Substance on the Public Health. T. L. Phipson. Chem. News, **59**, 255. Chloride of tin is used in preparing certain kinds of sugar for the market, as it gives them a golden hue which is much admired. The method the author used to detect the presence of tin in sugar is as follows. At least two ounces of the sample are taken, dissolved in cold water, and without separating the residue, the solution is made slightly acid with hydrochloric acid and then saturated with hydrogen sulphide. For two ounces of sugar about one pint of water and one to two drachms of hydrochloric acid were used. When the solution is saturated the bottle is covered with a plate of glass and the solution is allowed to deposit for at least forty eight hours. The clear liquid is then decanted off and the deposit is collected upon a filter and washed. The precipitate contains the sulphide of tin with insoluble impurities. The former is separated by washing with ammonium sulphide from which solution the tin is precipitated by adding first a sufficient amount of hydrochloric acid and the liquid is again allowed to stand for forty eight hours when the whole of the sulphide of tin is obtained, filtered and dried and transformed by calcination to stannic oxide and weighed. As to the toxic action of tin the author thinks that all sugars

in which a tin salt is used for curing and beautifying, should not be tolerated as it certainly is injurious. He also refers to baryta, strontia and oxalic acids, as other noxious substances proposed and used in sugar making. A. H.W.

On the Influence of Copper Upon the Determination of Iron in Ferro-Alloys.—T. W. Hogg, Chem. News **59**, 207. When an iron alloy is decomposed by dilute hydrochloric or sulphuric acid and then oxidized by means of a standard solution of potassium bichromate, if any copper is present it remains behind in the metallic state and reduces the iron, thus causing an error in the determination. To obviate the error, when copper or small quantities of organic matter are present the following method is given: a weighed portion of the alloy is dissolved in dilute hydrochloric acid, a slight excess of potassium chlorate is added, and the liquid is then well boiled to expel chlorine compounds. The small quantity of organic matter is then decomposed and the copper passes into solution. The iron is now reduced by means of sodium sulphite, and after the excess of sulphurous acid is expelled by boiling, the solution is titrated with standard potassium bichromate. A. H. W.

A New and Easy Method for the Rapid and Sure Detection of Mercury in Minerals.—Alexander Johnstone, Chem. News **59**, 221. When a small quantity of iodine newly liberated from a compound is allowed to impinge on metallic mercury, it instantly converts it into the characteristic scarlet mercuric iodide. The author gives the following table based on this reaction:

a. Heat the powdered substance in a small piece of hard glass tubing (say with a ¼ inch bore) closed at one end.

1. A metallic sublimate is produced. Proceed further by *b*.

2. A non-metallic sublimate is formed. Heat a fresh portion of the substance, mixed with about three times its bulk of fusion mixture ($K_2CO_3+Na_2CO_3$). A metallic sublimate forms. Proceed further by *b*.

3. There is no evidence of the slightest volatilization, other than water. No mercury is present in the mineral.

b. Pour into the tube containing the metallic sublimate and assay two drops of strong nitric acid, then at once one drop of

potassium iodide solution, and heat the bottom of the tube a little. A scarlet mass forms and remains on the sides of the tube. Mercury is present in the body tested.

When very minute quantities of mercury are present, proceed as follows: Place the mixture of powdered substance and flux in the closed tube and insert in the end a small piece of gold leaf on the end of a stout wire, which should be held just over the surface of the mixture, and apply heat for a minute or two. Any mercury, if present, will be volatilized, and will form a whitish amalgam with the gold. Now remove the wire with the gold leaf, touch the surface of the gold leaf which faced the assay with a single drop of concentrated nitric acid, and at once, on the top of this, add a drop of potassium iodide solution, when the unmistakable scarlet iodide will at once appear if any mercury were present in the body under examination. A. H. W.

On Cobalt and Nickel Peroxides and on the Volumetric Determination of these Metals.—Adolphe Carnot, Chem. News **59**, 183 (Comptes Rendus).

The author has made careful experiments to determine the state of oxidation of the precipitates of nickel and cobalt salts obtained by using potassium hydroxide, bromine, or an alkaline hypochlorite. He has found that the brown oxide obtained by precipitating cobalt with hydrogen dioxide and potassium hydroxide at a boil, has the exact composition of a sesquioxide, Co_2O_3. The black oxide of nickel, precipitated by hypochlorite or by bromine with potassium hydroxide, corresponds very nearly to Ni_2O_3. The cobalt oxide, nearly black, obtained by the use of hypochlorite, bromine, or iodine, always contains a proportion of oxygen decidedly greater than that in the sesquioxide. For example, with hypochlorite, the approximate formula is $Co_{10}O_{16}$, with bromine, and iodine, $Co_{10}O_{16.25}$. The author thinks a saline compound, containing a little cobalt dioxide along with sesquioxide, is formed in this case, with a probable formula $2CoO_2$, $4Co_2O_3$ for the hypochlorite precipitate, and $2CoO_2$, $3Co_2O_3$ for the bromine or iodine precipitate. It is certainly incorrect if we regard these precipitates as sesquioxides. Nickel may, however, be very well determined volumetrically, and a cobalt determination may be made accurately if hydrogen dioxide and

potassium hydroxide are used as reagents. If cobalt and nickel occur together, the cobalt can still be determined singly with accuracy, since the nickel is not peroxidized. A. H. W.

The Determination of Silicon in Commercial Aluminum.—A. J. Williams (a paper read before the Amer. Inst. Mining Engineers, New York meeting, Feb., 1889). The determination of silicon in aluminum by the usual method—that of solution in acid and evaporation to dryness—gives results which are necessarily too low, as the bluish black residue obtained after ignition, which is usually weighed as silica, contains also unoxidized graphitoidal silicon. In order to secure correct results it is necessary to fuse this residue with sodium carbonate, and to evaporate the hydrochloric acid solution of the fused mass once more to dryness. The silica obtained by ignition will then be white and completely oxidized.

The Separation of Cobalt and Nickel after Oxidation in an Ammoniacal Liquid.—Adolph Carnot, Chem. News **59**, 208. (Comptes Rendus, **108**, 741.) The author has studied the effects of bromine and of hydrogen dioxide upon cobalt salts in presence of ammoniacal salts and has experimented to separate nickel and cobalt from each other by one precipitation, by varying the state of combination of the cobalt, by peroxidizing by heat, or by heating the ammoniacal solution different lengths of time with sodium hypochlorite, bromine, or hydrogen dioxide, but without success. He found that the best results were obtained by leaving the solution for a few minutes at 100°, cooling, adding much water, and then a solution of potassium hydroxide; but the separation by the first precipitation was never satisfactory, the operation having to be repeated twice more, though the method may be recommended, because the presence of cobalt is shown by the rose coloration of the liquid as long as any traces of the cobalt remain. A. H. W.

A New, Easy and Rapid Method of Detecting Minute Quantities of Iron in Minerals.—Alexander Johnstone, Chem. News **59**, 231. Mix a little of the powdered mineral with about four times its bulk of potassium nitrate or chlorate, place on a clean piece of platinum foil and heat the platinum from below for a minute or two with a blowpipe flame. Before the ignited mass

has cooled, add by means of a glass tube, concentrated nitric acid drop by drop until only one drop remains not dried up, next add to the unevaporated nitric acid, two or three drops of an aqueous solution of potassium sulphocyanide, when the characteristic blood-red coloration will appear if any iron was present in the mineral under examination. A. H. W.

Method of Qualitative Analysis for the Separation and Detection of Al, Cr, Fe, Co, Ni, Mn, Zn, Ba, Ca, Sr, Mg, in the presence of Phosphoric, Arsenic, Boric, Silicic, Hydrofluoric, Acetic and Tartaric Acids and Organic Matter.—Wells and Vulte (School of Mines Quarterly, X, 255). The method is described as follows:

"If a solution containing the before mentioned acids and bases is made alkaline with ammonium hydrate, the following is likely to occur:

H_3PO_4	ppts Al Cr Fe… Co Ni Mn Zn Ba Sr Ca Mg
H_3BO_3	
H_4SiO_4	
HF	
$H_2C_2O_4$	chiefly
H_3AsO_4	Fe… and Mg
$H_2C_4H_4O_6$	prevent precipitation of Al, Cr, Fe, if boiling and neutral $HC_2H_3O_2$ throws down Al, Cr and Fe as Basic Acetates
$HC_2H_3O_2$	
Organic Matter	

"To test for the presence of these compounds three small portions of the main solution are taken as follows:

"Part I. Add HNO_3 and $(NH_4)_2MoO_4$; a yellow cryst. ppt. indicates phosphates, if heated gently a yellow color or ppt. indicates silicates and arsenates.

"Part II. Add $HC_2H_3O_2$ and NH_4OH in excess; a gelatinous ppt. indicates phosphates, borates, etc.

Part III. Add conc. H_2SO_4 and evaporate to dryness; a carbonaceous residue indicates organic acids or organic matter.

All the acids mentioned except phosphoric, boric and arsenic may be removed by evaporation with HNO_3 and ignition. Borates are soluble in a considerable excess of ammonium chloride and give no trouble.

Arsenates are removed by reduction with SO_2 and precipitation as As_2S_3 by H_2S. This should be done before adding HNO_3.

After making these tests, return to main solution.

"Boil out H_2S. Add a few drops HNO_3 (Note 1). If $H_2C_2O_4$ or organic matter is present evaporate to dryness and ignite gently. If not, evaporate, but do not ignite. Treat residue with HCl (conc.), dilute with H_2O and boil; it dissolves wholly or leaves a white residue of SiO_2. Filter. To filtrate add NH_4Cl and NH_4OH in slight excess, heat gently and filter quickly. Wash ppt. A with hot water. To filtrate add $(NH_4)_2S$ in slight excess, heat, filter and wash ppt. B. Examine filtrate for the alkalies and alkaline earths. Transfer ppts. A and B to a porcelain dish and digest with a little $(NH_4)_2S$. (Note 2), filter and wash with H_2O and $(NH_4)_2S$.

<table>
<tr><td rowspan="7">Filt.
$(NH_4)_3PO_4$
$(NH_4)_2S$
Reject</td><td colspan="6">Residue $Al_2(OH)_6$, $Cr_2(OH)_6$, FeS, $Al_2(PO_4)_2$, $Cr_2(PO_4)_2$, $Ba_3(PO_4)_2$, $Sr_3(PO_4)_2$ $Ca_3(PO_4)_2$ $NH_4Mg(PO_4)$, MnS, ZnS, CoS, NiS, S.
Treat with cold very dilute HCl, sp. gr. 1.05, filter and wash.</td></tr>
<tr><td rowspan="6">Residue
NiS+CoS+S
Test a portion in Borax Bead
Blue=Co
Brown=Ni
If blue, dissolve in Cl evaporate to expel xs acids and Cl, dil+H_2O nearly neutralize +KOH, add KCy until ppt. formed is dissolved, boil for 4 or 5 minutes, cool, add large xs KOH +Br water, a black ppt.= $Ni_2(OH)_6$, wash with very dilute NH_4OH to remove last trace of Co, test in Borax bead.
Brown Bead Ni</td><td colspan="5">Filtrate
Contains Fe, Al, Cr, Zn, Mn as chlorides, and Al, Cr, Ba, Sr, Ca, Mg as Phosphates.
Boil out H_2S, dilute and make alkaline with Na_2CO_3, add Bromine water and boil until Cr is all oxidized and Br. removed. Filter.</td></tr>
<tr><td rowspan="5">Filtrate
Na_2CrO_4.
Acidify with HA. Test for Cr with $BaCl_2$. yellow ppt. $BaCrO_4$ =Cr. (Note 3.)</td><td colspan="4">Ppt, $Al_2(OH)_6$, $Al_2(PO_4)_2$, $Fe_2(OH)_6$, $Fe_2(PO_4)_2$, Ba,Sr,Ca,Mg Phos.Zn,Mn Basic Carbs. Diss. in least possible quantity of dil. HCl and add Fe_2Cl_6 until a few drops of the sol. gives a yellow or red ppt. with NH_4OH. (Note 4.) Nearly neutralize with Na_2CO_3, dilute and add xs of NaA. Boil and filter hot. Wash ppt. with boiling H_2O.</td></tr>
<tr><td colspan="2">Ppt.
Fe and Al as Phosphates and Basic Acetates, add KOH and boil.</td><td colspan="2">Filtrate.
Zn, Mn, Ba, Sr, Ca, Mg as acetates or chlorides, add NH_4Cl, NH_4OH $(NH_4)_2S$. Filter and wash.</td></tr>
<tr><td rowspan="3">Ppt.
$Fe_2(OH)_6$
$Fe_2(PO_4)_2$</td><td rowspan="3">Filt.
Add HCl to acid reaction and then make alk. with $(NH_4)_2CO_3$ white ppt. $Al_2(OH)_6$ and $Al_2(PO_4)_2$</td><td>Ppt.
ZnS + MnS.</td><td>Filtrate Ba, Ca, Sr, Mg acetates or chlorides. Examine in usual manner.</td></tr>
<tr><td colspan="2">Treat with HA.</td></tr>
<tr><td>Res
ZnS
Treat on charcoal with $Co(NO_3)_2$ green mass Zn</td><td>Solution
MnA_2 add KOH white ppt. turning brown in the air, fuse with Na_2CO_3. Green mass Na_2MnO_4 indicates Mn</td></tr>
</table>

I. HNO_3 is added to oxidize Fe and destroy $H_2C_2O_4$, organic acids and organic matter.

II. NH_4OH ppts. Fe, Al, Cr as hydrates and phosphates, and the other metals of the group as phosphates, if they are in combination with that acid; it also ppts. all of the phosphates of the alkaline earths that may be present.

Ppt. A is treated with $(NH_4)_2S$ to convert the phosphates (except those of Al, Cr, Ba, Sr, Ca and Mg) into sulphides; the H_3PO_4 is taken up by the NH_4OH in a soluble form and filtered off, thereby making a partial separation of the phosphoric acid.

III. A pink or green coloration may be caused by the oxidation of Mn to permanganate or manganate, neither interfere with the $BaCl_2$ test.

IV. Ferric salt is added to carry down the phosphoric acid, as $Fe_2(PO_4)_2$, white, solution must be alkaline with NH_4OH; when all the H_3PO_4 is pptd. the NH_4OH brings down the yellow or brown $Fe_2(OH)_6$.

V. Instead of scheme as above the following may be used:

Filt. containing Mn, Zn, Ba, Sr, Ca, Mg as acetates or chlorides add Br and boil as long as a black ppt. is formed and color of Br remains; filter.

<table>
<tr><td rowspan="2">Ppt.
MnO_2H_2O
test in
Na_2CO_3
bead</td><td colspan="2">Filt. Zn, Ba, Sr, Ca, Mg acetates or chlorides.
Warm, pass a rapid current of H_2S, filter.</td></tr>
<tr><td>ZnS
white, if
dark colored
test on char-
coal with
$Co(NO_3)_2$.</td><td>Filt.
Boil out H_2S and proceed as usual.</td></tr>
</table>

"A scheme for separating and detecting H_2S, HCy, H_4FeCy_6 $H_6Fe_2Cy_{12}$, HCl, HI and HBr: Boil out H_2S, test fumes with PbA_2 paper, a black coloration indicates H_2S. Add $NaHCO_3$ and distill, carrying distillation to dryness, and pass distillate into KOH, test this for HCy in usual way. When cyanides are removed, cool, dissolve residue in H_2O and add $CuSO_4$ as long as a ppt. is formed, and until the liquid becomes blue or green; filter.

<table>
<tr><th>Ppt.</th><th>Solution.</th></tr>
<tr><td>Cu_2FeCy_6
$Cu_3Fe_2Cy_{12}$

NOTE.—Test for these acids in the original with Ferric and Ferrous salts.</td><td>Add conc. solution of $(NH_4)_2Fe_2(SO_4)_4$ and boil, pass vapor into starch water. Blue color indicates HI. When this is removed add a drop of conc. H_2SO_4, some dil. $K_2Mn_2O_8$, and boil as long as Br* is given off or until the color is discharged, adding more $K_2Mn_2O_8$ if necessary. Finally test soln. with HNO_3 and $AgNO_3$; a white curdy ppt. sol. in NH_4OH and pptd. by HNO_3 indicates HCl.</td></tr>
</table>

NOTE.—So much of the above scheme as relates to separation of Cl, Br, and I is due to E. Hart (See *Chem. News*, Dec., 1884). The use of $(NH_4)_2Fe_2(SO_4)_4$ is original."

The Enrichment of Phosphatic Chalk.—Phosphatic chalks when they are very pure can be enriched by washing until they may be made to contain from 30 per cent. to 40 per cent. of phosphate of lime. Dilute hydrochloric acid cannot be used for this purpose on account of the large amount of phosphate dissolved. On the contrary water charged with carbonic acid dissolves the carbonate of lime present very rapidly leaving almost pure the

*Br vapor may be passed into KOH and this solution tested in the usual way.

phosphate of lime. It is probable that the phosphatic deposits have been made in this way. The percentage of phosphate of lime in these chalks has been raised by such treatment from 15 per cent. to 60 per cent. (Nantier, Comptes Rendus, Vol. 108, No. 22, p. 1174.) H. W. W.

The Estimation of Sulphur in Burnt Pyrite and Analogous Substances.—G. Lunge, Ztschr, angew. Chem. 1889, 239. A review of the methods used by different chemists in estimating sulphur and a comparison of results obtained by these methods.

J. E. W.

The Estimation of Zinc with Ferrocyanide of Potassium in Ammoniacal Solutions.—A Voigt, Ztschr. angew. Chem. 1889, 307.

The material in which the zinc is to be determined is dissolved in hydrochloric acid, oxidized with nitric acid and rinsed into the titrating vessel with 100–150 cc. of water. 5 gms. to 10 gms. of tartrate of potassium are added to keep any iron in solution and the solution then made strongly ammoniacal and diluted with water to 250 cc. A solution of ferrocyanide of potassium is run into the solution from a burette, the end reaction being found by taking, from time to time, a drop of the solution from the dish and mixing with it a drop of concentrated acetic acid, until a blue color is obtained; this is best seen on a porcelain plate.

The ferrocyanide solution is made by dissolving 46 gms. of the clean commercial ferrocyanide of potassium in water, allowing it to stand for a day, and after filtering making up to one liter. For the standard solution of zinc, a solution of zinc oxide in hydrochloric acid is made, so that one liter contains 12.461 gms. of oxide; 10 cc. of this solution is diluted to 150 cc., 5 gms. of tartrate of potassium added, then a drop of ferric chloride, and after being made strongly ammoniacal, the whole diluted to 250 cc. and titrated with the ferrocyanide solution.

To obtain a fine blue color for the end reaction it is essential that there be no great excess of free ammonia present, otherwise the color will be of a greenish gray and indistinct.

If more or less than 10 cc. of the ferrocyanide solution is used the liquid is made stronger or more dilute as the case may be.

The determination is much quicker than the sulphide of sodium method, and the end reaction is clearly seen. Lead has no appreciable effect on the determination of the zinc; but the presence of much manganese causes the result to be too high.

J. K. W.

The Determination of Sulphuric Acid in Presence of Iron.—P. Jannasch and T. W. Richards, Chem. News **60**, 19 (and Journal fur praktische Chemie, New Series **39**, 321).

The authors of this article have experimented in a number of ways and have found that in presence of iron, an accurate determination of sulphuric acid by direct precipitation with barium chloride is not practicable. The only accurate method for pyrites is that of Fresenius, viz: fusing with a mixture of sodium carbonate and potassium nitrate and determining the sulphur in the filtrate from the water extract. The method of titration with barium chloride in presence of iron is unreliable, since the double sulphate of barium and iron is thrown down in this case, and too little barium chloride is consequently consumed.

A. H. W.

Determination of Silicon and Iron in Cryolite.—R. Fresenius and E. Hintz, Ztschr. anal. Chem. **28**, 324. When cryolite is decomposed with sulphuric acid only a small portion of the silica goes off as silicon tetrafluoride, most of it remaining in the residue. The determination is best made as follows: a **U** formed strong walled lead tube 18 cm. high and 2.6 cm. inside diameter is used as the decomposing vessel. This is filled with concentrated sulphuric acid and heated to 200° C. and then washed out carefully with water. This is done to cover the walls with a layer of lead sulphate. The tube is placed in a deep vessel filled with sand placed on a tripod so that it can be heated. The **U** tube is closed at both ends with rubber corks through which pass small leaden tubes. One of these tubes is connected with a washing flask containing sulphuric acid and the other with two similar leaden **U** tubes, slightly smaller (2 cm. wide). No glass can be used in making these connections, only lead and rubber. The last leaden **U** tube is connected with one made of glass and containing a little water, and this in turn with an aspirator. To conduct the analysis both leaden tubes are partly filled with ammonia, and about 6

gms. cryolite and 15 cc. sulphuric acid put into the decomposing vessel, which is heated to 200° in the sand bath for 2 hours while a slow stream of air is drawn through the apparatus. The contents of the tube are now washed into a platinum dish and heated with a few drops of hydrochloric acid to bring the alumina, etc. into solution. The solution is now filtered and the residue, which may still contain fluorides, treated for silica as follows: it is fused with sodium carbonate, dissolved in water, and hydrochloric acid added while the solution is hot, nearly but not quite to acid reaction, and the silica separated by evaporation. To the filtrate, a solution of zinc carbonate in ammonia is added and the solution evaporated until the ammonia is driven off. The silicon is separated from the precipitate in the usual way by dissolving it in nitric acid, evaporating, taking up with nitric acid and filtering off the silica. That part of the melt insoluble in water is dissolved in hydrochloric acid and the silica separated in the usual way.

The contents of the leaden tubes are now filtered and the residue consisting of silica and lead sulphate burned, the ash moistened with a few drops of nitric and sulphuric acids, ignited and the residue extracted with ammonium acetate, which dissolves the lead leaving the silica behind. The ammoniacal filtrate is treated with some sodium bicarbonate and the silica separated by addition of a solution of zinc carbonate in ammonia as before. The five lots of silica so separated are then united and weighed. Three different samples of cryolite treated as above gave 0.80, 1.23 and 1.39% silica.

The iron is determined by treating the cryolite with sulphuric acid as before and filtering off the insoluble residue. The residue is fused with sodium carbonate, evaporated with hydrochloric acid to separate silica and the filtrate added to the sulphuric acid solution; chlorine water is now added to oxidize the iron and the solution is poured into concentrated, hot, caustic potash solution. The small precipitate so obtained is well washed and dissolved in hydrochloric acid, tartaric acid added and the iron precipitated as sulphide by means of ammonia and ammonium sulphide, filtered off, converted into oxide as usual and weighed. In the three samples above mentioned 0.11, 0.85 and 0.88% iron was found.

Quantitative Determination of Thallium and Mercury.— W. Feit. Ztschr, anal. Chem. **28**, 314. When a thallic salt is added to potassium iodide, thallous iodide separates and iodine is liberated

$$Tl_2(SO_4)_3+6KI=2TlI,+3K_2SO_4+4I.$$

The iodine set free is determined by adding arsenious acid in excess and titrating back with iodine solution. The solution of arsenious acid is prepared by dissolving 4.942 gms. As_2O_3 in caustic alkali, neutralizing with HCl and diluting to a liter with sodium bicarbonate solution. Of this solution 1 cc.=.0100605 Tl. The iodine solution is about $\frac{n}{50}$. The thallium should be in the condition of thallic sulphate, and this may be effected by adding sufficient sulphuric acid and then bromine water until the solution is distinctly colored, and driving off the excess of bromine by boiling. The solution should then be treated with potassium iodide and made alkaline with sodium bicarbonate before the titration is proceeded with. If a decided excess of arsenious acid is added the change of color in the precipitate from greenish gray to the pure yellow of thallous iodide is rapid; in any event, this change will take place in fifteen minutes. The solution is now made up to a definite volume in a flask, filtered through a dry filter and an aliquot part titrated with iodine and starch solution. The method cannot be used when metals are present which act upon arsenious acid; such metals may, however, be first thrown down by means of sodium bicarbonate. Sodium thiosulphate may also be used instead of the arsenious acid. Six test analyses on pure thallium sulphate gave from 80.11 to 80.31 per cent. thallium instead of 81.12, theory. The same sample treated gravimetrically gave 79.56 %.

MERCURY.

If mercury salts are treated with an excess of sodium hydroxide and then with an alkaline solution of arsenious acid and boiled for some time, the mercury is entirely thrown down in the metallic form:

$$2HgO+As_2O_3=2Hg+As_2O_5$$
$$2Hg_2O+As_2O_3=4Hg+As_2O_5$$

If a mixture of mercurous and mercuric salt is at hand it is to

be converted into mercuric salt by boiling with excess of nitric acid, which, in the alkaline solution, is without influence upon the reaction. An excess of arsenic solution amounting to several cc. is added and the solution boiled 5–10 minutes, filtered through a dry filter, made up to the mark of the flask, an aliquot part taken, neutralized with HCl, sodium bicarbonate added and the excess of arsenious acid determined with iodine and starch. The mercury in salts containing chlorine cannot be determined by this method when it is necessary to first oxidize with nitric acid, since chlorine is set free and hypochlorites formed which act on the arsenious acid. It is not practicable to drive off the chlorine by evaporation since, according to Fresenius* mercury is volatilized. Mercuric sulphide also causes some trouble. In either of these cases the method used is as follows:

The substance, if it contains chlorine, is dissolved in hot nitric acid, if mercuric sulphide in aqua regia, an excess of sodium bicarbonate added, then potassium iodide until the precipitate redissolves, a slight excess of sodium arsenite is added, then a few drops of starch solution and now drop by drop iodine solution until the solution is colored blue. The remainder of the process is then finished as above indicated.

In a solution of mercuric chloride 10 cc. of which contained 0.1476 Hg. this method gave in 10 cc. 0.14847, 0.14747 and 0.14827. In 20 cc. 0.29795 and in 25 cc. 0.36948. In another solution of mercuric nitrate containing 0.1852 Hg in 20 cc., 20 cc. gave 0.18559 and 0.18489.

The Estimation of the Total Organic Nitrogen in Water by the Kjeldahl Process.—By Henry Leffmann and William Beam, Amer. Chem. Journ. **11**, 274.

The authors proceed as follows:

For nitrogen existing as ammonium.—200 cc. of the water to be tested are mixed with solutions of 2 cc. sodium carbonate and 2 cc. sodium hydrate. The solution is filtered through a clean cotton plug in a funnel, and after rejecting the first portion of the filtrate, 100 cc. are caught and nesslerized.

For total organic and ammoniacal nitrogen.—500 cc. of the water are boiled with 10 cc. concentrated sulphuric acid until

* Quant. Analyse 6, Aufl. Bd. 1, p. 322.

colorless or light yellow, potassium permanganate added and boiled until colorless; cooled. 50 cc. of water free from ammonia are added, about 150 cc. sodium hydrate solution, and 2 cc. sodium carbonate solution. Cooled, and then diluted to 250 cc. and set aside for the precipitate to settle. An aliquot part drawn off by means of a pipette and filtered through a cotton plug. Nesslerised and calculated as total nitrogen. A correction for impurity of reagents is determined by a blank experiment. C. W. M.

A Rapid Colorimetric Method of Determining Nitrates in Potable Waters.—Samuel C. Hooker, Amer. Chem. Journ. 1889, **11**, 249.* The green coloration produced by adding nitric acid to a sulphuric acid solution of carbazol has been used by Hooker to estimate nitric acid in potable waters. He concludes that:

"1. No material error will arise in the presence of nitrites in the quantities in which they usually occur.

2. Iron (in the ferrous or ferric condition), if present in quantities greater than 0.1 part per 100,000 must be removed.

3. Chlorides, even when present in very small quantities, must be removed:

4. Carbonate of lime, sulphates of lime, soda, etc., in the quantities in which they ordinarily occur, were not found to influence the determinations.

5. The presence of easily destructible organic matter, such as albumen, lowers the result; not materially, however, unless present in large excess."

Waters containing from .03 to .40 parts of N of nitrate per 100,000 can be tested directly on the original sample. Those containing more or less must be diluted or concentrated respectively.

The following reagents are used in the process:

1. Sulphuric acid of a sp. gr. of 1.84 free from oxides of nitrogen.

2. Carbazol dissolved in acetic acid (0.6 gm. in 100 cc.) glacial acetic acid.

3. A solution of carbazol in sulphuric acid; made by dissolving 1 cc. of carbazol in acetic acid in 15 cc. sulphuric acid 1.84. This solution may be used until six hours after preparation.

*See this Journal III, 69.

4. Several standard solutions of potassium nitrate are prepared from a dilute standard solution of nitrate. The standard solution is prepared by dissolving .7214 gms. potassium nitrate in one liter of distilled water. 1 cc.=.0001 gm. N. This solution is diluted ten times and forms the diluted standard stock solution. From this latter solution the various standards are made.

5. A solution of 5 gms. aluminum sulphate free from chlorine and iron in 1 liter distilled water.

6. A solution of sulphate of silver made by dissolving 4.3943 gms. of the salt in one liter of water. It may also be prepared by dissolving metallic silver in sulphuric acid.

The analysis is performed by taking 2 cc. of the water under examination in a test tube, adding 4 cc. sulphuric acid, cooling and adding 1 cc. of carbazol solution.

The depth of color is now imitated by means of the standard potassium nitrate solution by comparison.

Traces of iron and nitrites affect the results slightly. If chlorides are present they must be removed by the sulphate of silver solution. A little aluminum sulphate solution is also added to aid in the precipitation of the silver chloride. When the chlorides are removed the test is made as already described. C. W. M.

Determination of Zinc.—Max Bragard, Ztschr. anal. Chem. **27,** 209, has examined carefully the separation of zinc by means of sulphuretted hydrogen in presence of formic acid. He finds that the zinc is completely precipitated until the acid rises to 20 cc. (sp. gr. 1.1136) in 200 cc. solution containing 0.0325 gm. zinc. If the other conditions remain the same, the precipitation is more complete the greater the dilution. The absolute amount of zinc present has an influence; the same amount of acid which prevents the precipitation of small amounts will not prevent the precipitation of larger amounts. On the other hand, the larger the amount of zinc present, the larger the amount of formic acid set free, so that, for this reason, the amount of acid added cannot be increased in proportion. With 0.0325 zinc in 200 cc. liquid, not more than 15 cc. of formic acid of the above strength should be used.

The sulphuretted hydrogen should be passed through the cold solution and this then warmed. In this way the separation is

more rapid and the precipitate has the same properties as when thrown down from a hot solution.

Nickel may also be thrown down completely from a dilute formic acid solution by means of sulphuretted hydrogen. The precipitation is more complete from a warm solution, especially on long standing, than from a cold solution. The concentration of the solution appears, in this case, to be without influence upon the precipitation. With a large excess of formic acid (5 cc. acid to 0.0240 nickel) the nickel is not thrown down. For the separation of zinc from nickel the author therefore gives the following rules:

1. The solution must be sufficiently acid to prevent precipitation of the nickel (5 cc. formic acid, sp. gr. 1.1136 to 0.03 gm. nickel).

2. The free acid should not exceed a certain amount or the zinc will not be completely thrown down. If this is unavoidable (as in presence of much nickel) then

3. The solution must be sufficiently dilute (500–600 cc).

4. If precipitation takes place in a hot solution, more acid is necessary than in the cold.

The method as outlined above generally gives a sulphide which is more or less colored from presence of nickel sulphide. In this case it is necessary to repeat the precipitation by dissolving the sulphide in hydrochloric acid, driving off the sulphuretted hydrogen, adding ammonia to neutralization, then formic acid and repeating the precipitation with sulphuretted hydrogen.

No.	Zn.	Ni.	Vol. in cc.	CH_2O_2 in cc.	In 1st filt. Ni.	ZnS found.	= Zinc.	Ni in 2d filt.	Ni pptd. in 1st ppt.
1	0.0325	0.0048	300	5	0.0023	0.0483	0.0324	0.0026	54.2
2	"	"	"	6	0.0029	0.0485	0.0325	0.0018	37.5
3	"	"	"	7	0.0036	0.0480	0.0322	0.0011	22.9
4*	"	"	250	7	0.0048	0.0488	0.0327	. .	. .
5	"	"	300	10	0.0049	0.0486	0.0325	. .	. .
6	"	0.0096	500	"	0.0096	0.0484	0.0325	. .	. .
7	"	0.0240	"	"	0.0236	0.0483	0.0324	0.0005	2.1
8*	"	0.0480	"	"	0.0482	0.0488	0.0327	. .	. .
9	0.3250	0.0960	400	"	0.0691	0.4853	0.3252	0.0270	28.1
10	"	"	500	20	0.0782	0.4846	0.3247	0.0177	18.4
11	"	"	600	30	0.0885	0.4851	0.3250	0.0077	8.0
12	"	"	"	40	0.0959	0.4847	0.3248	. .	. .

* In 4 and 8 the precipitation took place in the cold, in all other cases the solution was hot.

With the same precautions zinc may be separated from iron; in this case, however, since iron is more easily thrown down than nickel, a larger amount of acid must be added and the dilution increased in proportion.

No	Zn.	Fe.	CH_2O_2 in cc.	Vol. in cc.	Iron in ZnS.	In % of the iron present.
1	0.0318	0.0072	5	300	0.00110	15.3
2	"	"	6	"	0.00083	11.5
3	"	"	7	"	0.00056	7.8
4	"	"	10	400	0.00026	3.6
5	"	0.0360	"	"	0.00028	0.8
6	0.0636	"	"	"	0.00032	0.9
7	"	"	15	"	. .	. .
8	0.6360	0.3600	"	500	0.00071	0.2
9	"	0.7200	17	"	0.00012	0.02
10	"	"	20	"	. .	. .

The method of Beilstein,* according to which zinc is thrown down from a citric acid solution is not a desirable one, because, although the separation from nickel is complete, the precipitated zinc sulphide is, like that thrown down by ammonium sulphide, difficult to filter and wash.

Bragard has also tested Tamm's† method, in which the zinc is precipitated as zinc ammonium phosphate in presence of ammonium chloride. The precipitation is most complete when 400 cc. of solution contain 0.2–0.4 gm. zinc, and when the precipitate is allowed to stand 24 hours before filtering. In order to prevent the precipitate from running through the filter, it is best to wash with water of the same temperature as the filtrate at first, gradually heating it as the washing proceeds. Tamm directs that the precipitate shall be dried at 100° and weighed. In order to avoid this tedious operation, Bragard filters on a small filter, separates it as completely as possible after drying, and ignites the precipitate carefully, taking care not to raise the temperature high enough to melt it, and to exclude reducing gases from the cruci-

* Ztschr. anal. Chem. **18**, 262.

† Ibid. **12**, 320.

ble. The filter is either ignited carefully with addition of nitric acid, or it is treated with dilute nitric acid to dissolve the adhering precipitate, and the filtrate added to the main portion, and the two evaporated to dryness and ignited together.

The statement of Lösekann and Meyer* that zinc may be separated from magnesia by means of sodium phosphate and excess of ammonia, but not from manganese, the author finds to be correct, provided that too much magnesia is not present, while manganese is thrown down free from zinc only if a small amount is present.

Neumann (Ztschr. anal. Chem. **28**, 57) has examined the separation of zinc from manganese in formic acid solution and finds the separation to be satisfactory.†

Detection of Nitrous Acid in Potable Water.—Instead of distilling the water to remove the bacteria as Musset‡ describes, Proskauer (Chem. Zeit. Rep. **13**, 146) simply filters the water through a thick, well washed filter paper and uses the iodine, zinc, and starch solution test. The bacteria will remain on the filter. He also recommends the test with metaphenylendiamin. S. C.

Detection of Tantalum and Niobium.—K. Haushofer (Chem. Zeit. Rep. **13**, 133). The mineral is heated with concentrated sulphuric acid, and after separating the insoluble residue the solution is diluted with water and some zinc dust is added. In a few minutes the liquid assumes a sapphire blue color which gradually changes to an olive green or violet and a fine white powder separates out. S. C.

Determination of Cadmium and its Separation from Copper.—A Kohner, Ztschr. anal. Chem. **27**, 213. 1. The determination of cadmium as oxide gives too low results because it is so easily reduced. 2. The method of Carnot and Proromont§ (determination as pyrophosphate) is inexact. 3. The cadmium can not be weighed as sulphide, because sulphur is precipitated with it. For technical purposes this methed may, however, be sometimes useful. It should be precipitated at 50°, washed, after drying, with carbon disulphide or sodium sulphite, and then

* Chem. Ztg. **10**, 729.

† See also this Journal **1**, 208.

‡ This Journal **3**, 198.

§ Comptes rendus **101**, 59.

dried at 115°. The sulphide may also be treated with hydrochloric acid and the sulphuretted hydrogen given off titrated with iodine solution. This method gives excellent results for small amounts (0.07–0.6 gm. Cd). 4. The determination as sulphate gives good results with large, but not with small quantities, since with small amounts decomposition is apt to take place on heating.

The author has also examined the following methods for the separating of cadmium from copper:

a. Treatment of the Sulphides with Sulphuric Acid.—The method, proposed by A. W. Hofmann,* was found to give correct results only when carried out as follows: After both metals have been precipitated from a neutral solution with sulphuretted hydrogen, a quantity of sulphuric acid is added proportioned to the water present, the solution boiled two hours and filtered from the crystalline copper sulphide. This filtrate is then precipitated with sulphuretted hydrogen, after being diluted with much water, and the precipitate, after filtration, treated on the filter with warm hydrochloric acid. A small amount of copper sulphide remains upon the filter and is to be added to the main amount.

b. Separation by Potassium Iodide.†—This method (separation of the copper as Cu_2I_2 with KI and H_2SO_3) can only be used when the amount of copper present is known approximately, since either potassium iodide or hydriodic acid in excess dissolve cuprous iodide.

c. Separation with Glycerol-soda lye, and with an alkaline Solution of Sodium Tartrate.—Donath‡ has described a method in which glycerol-soda lye is used to precipitate the cadmium, the copper remaining in solution. According to Kohner there are two objections to the method. First, the cadmium hydroxide carries down copper which cannot be separated by washing, and, second, the viscosity of the solution makes it difficult to wash the cadmium hydroxide free from alkali.

An analogous method proposed by Behal§ in which an alkaline solution of sodium tartrate is used gave better results; but the complete separation of the alkali causes much loss of time.

* Ann. Chem. (Liebig) **115**, 286.
† Compt. rend. **47**, 294; J. prakt. Chem. **61**, 105.
‡ Ding. poly. J. **229**, 542.
§ J. de Pharm. et de Chim. [Série 5] **11**, 553.

d. Separation with Ammonium Benzoate.—Gucci* proposes to throw down the copper as benzoate, using a 10 per cent. solution of ammonium benzoate. This method gives good results only when the solutions are concentrated and the precipitate is washed with a solution of ammonium benzoate. The ignition of this precipitate is attended with considerable difficulty. The methods in which organic substances are used for the separation of cadmium from copper offer no advantages.

e. Separation with Sodium Thiosulphate.†—This method gave imperfect results, since cadmium sulphide is always precipitated with the copper sulphide in small quantity.

On the Detection and Estimation of Caustic Alkali in Presence of Alkaline Carbonates.—L. Dobbin, J. Soc Chem Ind. **7**, 829.

The author makes a testing solution by dissolving 5 gms of potassium iodide in water, adding mercuric chloride until a permanent precipitate is obtained, then adding 1.3375 gms of ammonium chloride, and making up to one liter. On keeping a small quantity of red precipitate settles to the bottom. The reagent gives no coloration or precipitate on the addition of sodium or potassium carbonate, but it forms a delicate test for caustic alkali when used as follows. A standard solution of caustic soda free from carbonate is prepared by dissolving 2 [illegible] gms of sodium in recently boiled and cooled distilled water and making up to one liter. Then into a narrow flat bottomed test tube put the solution to be tested. To this is added 5 cc. of the standard ammonium chloride and double iodide solution, and its color compared with one produced by the standard caustic soda solution containing a known amount of alkali. The alkali is then easily calculated. The presence of alkaline carbonates or sulphates does not interfere with the estimation. This method the author states is not applicable to solutions which contain sulphides, as it interferes with the coloration. A very delicate qualitative reagent for detecting caustic alkalies may be prepared by adding potassium iodide to a mercuric chloride solution [illegible] precipitate first redissolves and adding a drop [illegible]

[illegible]

Determination of Tungsten in Alloys.—Preusser, Ztschr. anal. Chem. **28,** 173. 0.5–1 gm. of the alloy, which has been powdered in a steel mortar and bolted through linen, is placed in a porcelain dish and roasted over a Bunsen burner or in a muffle until the color changes to a pure yellow. The powder is then treated with aqua regia and evaporated to dryness to complete the oxidation, and then evaporated several times with hydrochloric acid to dryness and heated to 120° in the air bath. The mass is then treated with dilute acid, allowed to stand several hours and filtered. The residue which contains the silica and tungstic acid is intimately mixed with 3–4 times as much sodium carbonate, and fused half an hour over the blast lamp. The mass is then treated with hot water, filtered, and any tungstic acid remaining again fused with sodium carbonate. The filtrate, which contains the tungsten as sodium tungstate, is treated with hydrochloric acid, which throws down most of the tungstic acid as a white hydrate. To complete the separation the filtrate must be evaporated to dryness with the addition of acid. The residue is then treated with water, allowed to stand several hours and filtered with the aid of a filter pump. The residue is best washed with a dilute solution of ammonium nitrate. The precipitate, together with the filter, is brought into an Erlenmeyer flask, the tungstic acid which adheres to the dish is dissolved in ammonia and poured into the flask, more ammonia is added, and the solution gently heated. This causes the tungstic acid to dissolve while the silica, being nearly insoluble,* remains behind. The filtrate is evaporated to dryness in a weighed porcelain crucible and ignited to constant weight.

The method, adopted in some laboratories, of separating silica from tungstic acid by means of potassium carbonate, gives inexact results, since in the cold the silica is only partly dissolved, and when heated tunstic acid goes in solution. If tin is present, the method of Donath and Müller† should be used. The alloy is roasted, evaporated with aqua regia, heated to 120°, extracted with dilute acid, and the process completed according to the above directions.

*See Ztschr. anal. Chem. **11,** 182.
† This Journal II, 190.

Determination of Water and Carbon Dioxide in Salts.—T. M. Chatard, Am. J. Sci. [3], **37**, 468. A short piece of combustion tube is drawn out at one end into a smaller tube, which is bent at a right angle and fitted through the cork of a **U** tube. The **U** tube is made of small tube in the bend, in the middle of which a good sized bulb is blown, which is filled with sulphuric acid. Both legs of the **U** tube contain glass beads wetted with sulphuric acid. The substance (about 1 gm.) is placed in a platinum boat and pushed into the open end of the combustion tube, resting upon a piece of thin asbestos paper to prevent adhesion to the glass. A roll of asbestos paper, loosely filling the tube, is then pushed in, the tube corked, and a current of dry air drawn through the apparatus while the tube is heated. At the end of the operation water is apt to condense in the upper end of that leg of the **U** tube next to the combustion tube. In order to prevent this a small water bath is used having a ⊐ shaped recess in one side. This is fitted over the end of the tube and heated by means of an alcohol lamp. The gain in weight of the **U** tube is the water present. If the combustion tube is weighed before and after, the loss of weight $= H_2O + CO_2$, and by subtracting the H_2O found, the weight of CO_2 present may be determined.

Notes on the Electrolytic Assay of Copper.—Wm. Glenn (a paper read before the Amer. Inst. Min. Eng., Feb., 1889). The author uses a solution prepared as follows:

"Upon 2 grammes of any copper-bearing product (except bar-copper), placed in a beaker of 2 in. diam. and 5¼ in. depth, is poured at once a mixture of 3 cc. concentrated sulphuric acid and 17 cc. concentrated nitric acid. The beaker is then quickly covered and set upon a cast-iron plate 5/8 in. thick, under which is a lighted (really good) Bunsen burner, care being taken that the beaker be not nearer the flame than six inches, because, otherwise, sulphur liberated and fused might spoil the assay. When all turbulent action has ceased, the beaker is uncovered, placed four inches from the flame, and permitted to evaporate to dryness. It is then moved immediately over the flame, and left until SO_3 fumes come off freely. One must here be certain that no nitric acid remains, or it may happen, as it did with sample No. 5 of the series given below, that considerable copper is left in solution

after electrolysis. After treatment as mentioned, the mass within the beaker will always be found spongy, without sulphur-balls, and at no time will bumping or spurting occur. To the cool mass, about 60 cc. of water may be added, and the vessel returned to the hot plate, to heat and boil a half-hour. Filter into a 100 cc. measuring-flask; make up to the mark; and, by means of a 50 cc. pipette, transfer in equal portions into two 100 cc. beakers. Electrolyze both solutions, using cylinders 3.3 by 3.8 cm., well covered by the solutions, made up with water. For the other terminal, employ preferably a cylinder of 2 cm. diameter.

"Of course, one may electrolyze but a single solution; but in that case the flask and pipette must be in agreement, and the measuring accurately performed.

"If the work be important, after the copper-plated platinum cylinders are withdrawn for weighing, both residues should be poured together, heated to boiling, removed from the flame, and treated with a rapid stream of H_2S.

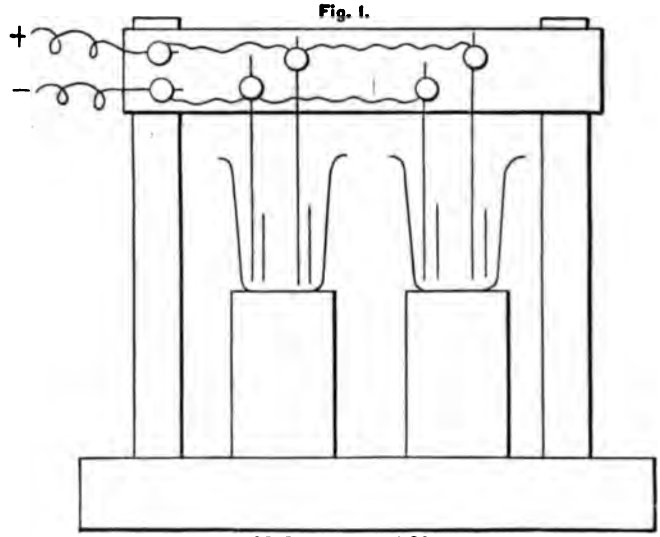
"The precipitated sulphide may be filtered off, ignited in the filter wrapped by a wire, the ash dissolved in 2.5 cc. nitric acid, made alkaline by 5 cc. ammonia water, filtered, and made up to 10 cc. with wash-water. The copper is estimated with KCy of the strength of 6 grammes to the liter of water. Below are given five determinations from a random page of the writer's note-book, showing what was found on the two cylinders, and what in the two residues combined.

No.	Kind of Material.	Copper on No. 1 Cylinder.	Copper on No. 2 Cylinder.	Found in residues of Electrolysis.	Per cent. of copper in sample.	Quantity used for assay.
1	Bar-Copper	.7352	.7335	.0007	97.78	1-20 of 15.028 gms.
2	Matte	.2221	.2216	.0006	44.42	1 gm. for both assays.
3	Sulphide ore	.1204	.1212	.0005	12.10	2 gms. for both assays.
4	"	.1210	.1212	.0010	12.16	"
5	Matte	.4573	.4562	.0033	45.84	"

The battery preferred is a large Bunsen cell of 1 gallon capacity charged with water in the outer cell to the depth of 7–8

cm., and the porous cup filled with a mixture of sulphuric acid (1.47 sp. gr.) 1 liter; water, 1 liter; sodium bichromate, 200 gms. An equal weight of potassium bichromate may be used, but the resulting salts are more likely to crystallize out than the sodium salts. 500 cc. of this solution will fill the cell. The stand used for precipitating the copper is shown in the figure.

Fig. 1.



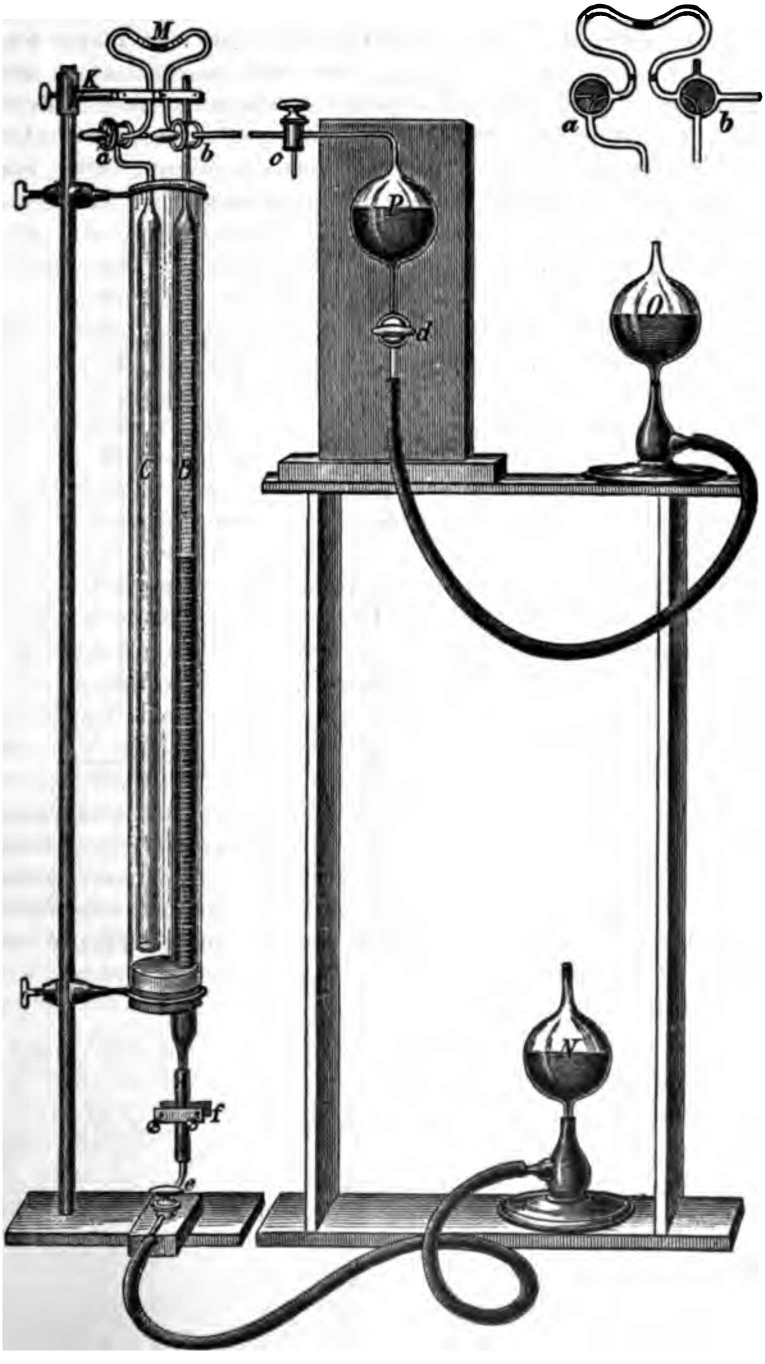
FRAME FOR ELECTROLYTIC COPPER-ASSAYS.

It is made of pine, and is shown in one-quarter its natural size. The author prefers beakers to platinum dishes. The objections to dishes are that an oxide (perhaps ferric oxide) is sometimes deposited and weighed as copper, and also that as evaporation takes place the copper is exposed in part to the oxidizing action of the air.

GAS ANALYSIS.

Contributions to Gas Analysis.—H. Drehschmidt, Ber. **21**, 3242. *An Apparatus for Gas Analysis.*—The apparatus of Hempel* has the disadvantage that it is not easy to tell when the mercury is at the same height in both legs of the manometer. If the temperature and pressure of the atmosphere have changed, the

*This Journal II, 84.


M
K
a
b
c
P
d
O
C
B
f
e
N
a
b

rubber connection between compensation tube and manometer must be opened and closed again to avoid inconvenience. The changing of gases in the longer leg of the manometer leads to errors if the burette is nearly full of gas, as it is necessary to sink the pressure bulb so far that gas enters the rubber connection and part of it may remain there. To avoid these difficulties the author has modified the apparatus as shown in the cut. His modification follows more closely the plan of Petterson's apparatus.*

The measuring tube *B* is connected below to the stopcock *c* and pressure bulb *N*. The clamp *l* allows of an exact adjustment of the height of the mercury. By means of *b* it may be connected with the manometer *M* or with the pipette *P*. The compensation tube *C* has a three way cock *a*, by means of which it may be connected with *B* or with the outside air. *C*, *M* and *B* are not blown in one piece, but are connected by means of short pieces of rubber tube not shown in the cut. The gas is measured as directed by Petterson.

The pipette used by the author differs somewhat from that of Hempel. The three way cock *c* has a capillary bore so that gases may be passed into *B*, and reagents may be drawn through the point into *P* by lowering *O* and opening *d*.

2. *Burning Gases without Explosion.* For burning hydrogen palladium asbestos, as directed by Winkler, was used. In order to prevent danger of explosion, pieces of platinum wire which nearly fill the tube are placed on both sides of the asbestos. Methane is burned by mixing with pure oxygen and passing through a platinum tube [illegible] cm long and 2 cm outside and [illegible] inside diameter. The bore of the tube is nearly closed by a palladium wire which passes through it. The middle part of the tube is heated to bright redness for a length of 6-7 cm, and the gas passed slowly back and forth two to three times.

Contributions to Gas Analysis. Cl. Winkler (Ztschr. anal. Chem. **28**, [illegible]).

[illegible] *Carbon Monoxide.* [illegible]

Hempel [illegible] absorbing carbon mon[illegible] [illegible] increase resulted. The [illegible]

[illegible] **28**, [illegible]

he explained by the supposition that ethylene is also absorbed and again given off if the solution is used several times. This explanation was disputed by Drehschmidt,* who found that the increase in volume occurs in the entire absence of heavy hydrocarbons. He attributed this result entirely to the loose state of combination in which the monoxide is held by the cuprous chloride, it being absorbed from gases rich in monoxide and given up in part to gases poor in monoxide introduced into the absorbing tube subsequently. This is especially noticeable with the hydrochloric acid solution; in ammoniacal solution it is much less marked, perhaps owing to the slow conversion of the monoxide to dioxide with separation of metallic copper. According to Drehschmidt,† two separate solutions of ammoniacal cuprous chloride should be used for the complete absorption of carbon monoxide, the first of which removes the greater portion, and the second, a fresh solution, completes the absorption and may be used again as solution No. 1 in a subsequent analysis. Winkler confirms Drehschmidt's observations in every particular. It would seem to be necessary to free the gas from ammonia after treatment with ammoniacal cuprous chloride before measuring, and this may be done in a separate pipette with dilute sulphuric acid. To avoid this, Winkler has endeavored to find an organic substance, which added to the acid cuprous chloride would prevent its precipitation by potassium hydroxide in excess, but so far without success.

Leybold‡ finds that cuprous chloride solution which has absorbed oxygen by exposure to the air may be made to give off the absorbed oxygen by exposure to carbon monoxide; but Winkler thinks this to be an error into which Leybold fell through determining the oxygen in illuminating gas by means of phosphorus which does not always absorb all the oxygen. When alkaline pyrogallate was used instead, no such result could be obtained.

Detection of Carbon Monoxide.—When carbon monoxide is brought in contact with palladious chloride solution it is very slowly absorbed and palladium separates. The reaction is much more satisfactory and prompt if a solution of cuprous chloride in a saturated salt solution, to which a few drops of hydrochloric acid

*Ber. **20**, 2752.
†Ber. **21**, 2158.
‡Chem-Ztg. 1888, 1277.

have been added, be used for absorbing the carbon monoxide. A few drops of a solution of sodium palladious chloride is then added, which causes the precipitation of black metallic palladium, the carbon monoxide being converted into dioxide. Before adding the sodium palladious chloride it is best to dilute the cuprous chloride solution with four times as much water. As little as .01 cc. carbon monoxide = 0.0000125 gm. may be detected in this way. A mixture of cuprous and palladious chlorides dissolved in a solution of common salt may also be used for the quantitative determination of carbon monoxide with excellent results. Other gases, however, are also absorbed to some degree, and oxidized by the solution, as, for example, hydrogen.

Determination of the Heavy Hydrocarbons.—Drehschmidt* and Treadwell and Stokes† find that bromine and nitric acid cannot be used for the separation of ethylene from benzene in presence of carbon monoxide, and that only the "total heavy hydrocarbons" can be determined by means of fuming sulphuric acid. The author's observations confirm these statements.

Determination of Methane.—In illuminating gas, for example, carbon dioxide is first determined with caustic potash, heavy hydrocarbons by means of fuming sulphuric acid, oxygen with alkaline pyrogallate and carbon monoxide with cuprous chloride. The remaining gas is then mixed with more than sufficient air to ensure complete combustion of the hydrogen, and the mixture led over slightly heated palladium asbestos. The remaining gas consists of methane, nitrogen and excess of air. Enough air is now added, so that for each volume of methane there are at least two volumes of oxygen and the gas is slowly led into a Hempel's pipette in which a platinum spiral is heated to redness. This pipette is made by inserting electrodes through a tubulure at the bottom closed with a cork. The electrodes pass into the pipette almost to the top where they are connected by a platinum spiral. The mixture of gases if run in slowly burns quietly; but if run in too rapidly an explosion ensues.

*Post's chem. techn. Analyse, 2 aufl., Bd. 1, Lief 1, p. 108.
†This Journal III, 94.

ATOMIC WEIGHTS.

EDITED BY F. W. CLARKE.

Oxygen.—Noyes has sought to determine the ratio of oxygen to hydrogen by passing the latter gas over hot copper oxide, in an apparatus so constructed that the water formed is retained in the same apparatus. The increase in weight of the latter gives the amount of hydrogen absorbed. The oxygen was then found by expelling the water which had been formed, and determining the loss of weight. The corrected weights are as follows:

.9443 H unites with 7.4976 O.		O = 15.880.
.6744 " " " 5.3551 "		" 15.881.
.7866 " " " 6.2554 "		" 15.905.
.5521 " " " 4.3856 "		" 15.887.
.4274 " " " 3.3927 "		" 15.876.
.8265 " " " 6.5658 "		" 15.888.

In mean, $O \doteq 15.886, \pm .0028$. Noyes is inclined to regard this value as affected by some undiscovered constant error (*Am. Chem. Journ.* **11**, 155.)

In the *Chemical News* for June 7, 1889, the foregoing determination is criticised by G. Stillingfleet Johnson, who points out two sources of error. One is the occlusion of hydrogen by the copper, the other is the adhesion of water. He also suggests the possible presence of sulphur in the copper oxide employed. Johnson cites in evidence a paper of his own which appeared in the *Journ. Chem. Soc.* for May, 1879.

Lord Rayleigh (Chem. News **59,** 147) has also published a preliminary note upon the same constant. Weighed quantities of oxygen and hydrogen were burned together, and the residue was analyzed. Five determinations gave for O the values

15.93
15.98
15.98
15.93
15.92

Mean, 15.95

Corrected for difference in volume between the full and the empty globes in which the gases were weighed, this becomes $O = 15.89$.

Zinc.—To determine the atomic weight of zinc, Gladstone and Hibbert compare the amount of zinc dissolved in a voltameter with the amounts of silver or copper precipitated by the same current. The following weights are given, in grammes:

.7767	Zn =	2.5589	Ag;	or	$\frac{Ag}{Zn}$ =	3.2945
.7758	"	"	"		"	3.2984
.5927	"	1.9551	"		"	3.2986
.5924	"	"	"		"	3.3003
.2277	"	.7517	"		"	3.2969
.2281	"	"	"		"	3.2955
.7452	"	2.4588	"		"	3.2995
.7475	"	"	"		"	3.2894
.8770	"	2.9000	"		"	3.3067
.8784	"	"	"		"	3.3015
.9341	"	3.0809	"		"	3.2982
.9347	"	"	"		"	3.2961
					Mean,	3.2981

Hence, if Ag = 107.66, Zn = 65.29.

.7667	Zn =	0.7526	Cu;	or	$\frac{Zn}{Cu}$ =	1.0313
.7758	"	"	"		"	1.0308
.5927	"	.5737	"		"	1.0331
.5924	"	"	"		"	1.0326
.2277	"	.2209	"		"	1.0308
.2281	"	"	"		"	1.0326
.8770	"	.8510	"		"	1.0305
.8784	"	"	"		"	1.0322
.9341	"	.9038	"		"	1.0336
.9347	"	"	"		"	1.0342
					Mean,	1.0322

Hence, if Cu = 63.33, Zn = 65.37. This is probably a trifle too high, and 65.3 is not far from the true value.—*Journ. Chem. Soc.* **55**, 443.

Chromium.—Ammonium dichromate has been used by S. G. Rawson in an investigation upon the atomic weight of chromium. The salt, purified by repeated crystillizations, was treated in a platinum dish with hydrochloric acid and alcohol, and the mass was then evaporated to dryness over a water-bath. After repeat-

ing this treatment, to ensure complete reduction, the mass was dissolved in water and ammonia was added. After evaporation to dryness the mass was heated in an air-bath for about five hours to 140° and finally ignited strongly in a gas muffle. The residue, Cr_2O_3, was weighed, and all the weighings were reduced to vacuum standards. At no point in the process was there any transfer of material from one vessel to another. The following results were obtained, assuming H = 1, N = 14.02, and O = 15.96.

$Am_2Cr_2O_7$	Cr_2O_3	At. Wt. Cr.
1.01275 gms.	0.61134 gms.	52.130
1.08181	.65266	52.010
1.29430	.78090	52.020
1.13966	.68799	52.129
.98778	.59595	52.016
1.14319	.68987	52.059
	Mean,	52.061
	Maximum difference,	0.120

Jour. Chem. Soc. **55**, 213.

Zirconium.—Bailey, after prolonged investigation of various zirconium compounds, has used the sulphate as the salt best suited to an atomic weight determination. Upon ignition, the compound Zr $(SO_4)_2$ yields ZrO_2. Bailey's data are as follows:

2.02357	gms.	sulphate	gave	0.87785	ZrO_2.	43.381
2.6185	"	"	"	1.1354	"	43.360
2.27709	"	"	"	.98713	"	43.350
2.21645	"	"	"	.96152	"	43.385
1.75358	"	"	"	.76107	"	43.402
1.64065	"	"	"	.7120	"	43.397
2.33255	"	"	"	1.01143	"	43.361
1.81105	"	"	"	.78485	"	43.337
					Mean,	43.37[illegible]

Hence, if H = 1, Zr = 90.401 in mean.—*Proc. Roy. Soc.* **46**, 74.

Tellurium.—During the past six years Brauner has been at work upon the atomic weight of this metal, which is peculiarly interesting as an apparent exception to the periodic law. Theoretically, its atomic weight should be below that of iodine; but all determinations hitherto have made it higher. Brauner made experiments with many tellurium compounds, the distilled bro-

mide, $TeBr_4$ being apparently the best. With this salt, four careful titrations with pure silver gave in mean Te = 127.61, a value far in excess of I, 126.86.

In order to explain this anomaly, Brauner has made some careful fractionations of the bromide, and has also determined the atomic weight of Te from the dibromide, $TeBr_2$. With *undoubted* $TeBr_4$ he obtained values for Te ranging as high as 127.72, and with the dibromide as high as 133. He concludes that the tellurium hitherto known is not a simple substance, but a mixture of probably three elements, and that true tellurium is yet to be discovered. *Jour. Chem. Soc.* **55**, 382.

Nickel and Cobalt. —Winkler's paper upon the atomic weights of these metals is essentially a discussion of methods previously used by him, in connection with the recent investigations of Krüss and Schmidt. He points out sources of error in the work of the latter chemists, but gives no new determinations of the constants. *Berichte* **22**, [illegible]

Ruthenium. Joly (Compt. rend. **108**, 946) determines the atomic weight of this metal by the reduction of its compounds in hydrogen. With the oxide RuO_2, he gives the following results:

[illegible]	oxide gave	[illegible]	of metal	or	[illegible]
[illegible]		[illegible]			[illegible]
[illegible]		[illegible]			[illegible]
[illegible]		[illegible]			[illegible]
				Mean	[illegible]

Hence if H = 1, Ru = 101.41; if O = 16, Ru = 101.[illegible]

With the compound $RuCl_3NO.H_2O$ similarly treated the percentage of ruthenium in two experiments was found to be [illegible] and [illegible]; mean [illegible].

Hence [illegible] H = 1, O = [illegible], N = 14.0[illegible], and Cl = 35.[illegible], Ru = [illegible]

The [illegible] salt $Ru_2Cl_3(NO)_2.2NH_4Cl$ gave [illegible] and [illegible] per cent [illegible]; mean [illegible]. Hence Ru = 101.[illegible]. Probably [illegible] the true value for ruthenium.

Palladium. [illegible] Keiser, who ignited palladium [illegible] purified in a stream of hydrogen

The formular of the salt is $Pd(NH_3Cl)_2$, and the weights are as follows :

	.83260	salt gave	.41965	Pd.
	1.72635	" "	.86992	"
	1.40280	" "	.70670	"
	1.57940	" "	.79562	"
	1.89895	" "	.95650	"
	1.48065	" "	.74570	"
	1.56015	" "	.78585	"
	1.82658	" "	.92003	"
	2.40125	" "	1.20970	"
	1.10400	" "	.55629	"
	.93310	" "	.47010	"
Sum,	16.74583		8.43606	

Hence, if H=1, N=14.01, and Cl=35.37, Pd=106.552. This is from the sum of the weights ; the separate experiments giving a maximum of 106.459 and a minimum of 106.292.

In order to prevent error due to occlusion of hydrogen by the residual palladium, the latter was allowed to cool finally in a stream of dry air.—*Proc. Chem. Section of the Franklin Institute, for the meeting of March 19, 1889.*

Gold.—Mallet, in seven distinct series of experiments, has redetermined the atomic weight of gold. His mean result is Au=196.910. The complete paper, with details, is yet to appear.—*Abstract in Proc. Royal Soc.* **46,** 71.

ORGANIC ANALYSIS.

Simultaneous Determination of Hydrogen and Nitrogen.—C. Gehrenbeck, Ber. **22,** 1694. This method supplements the method of Messinger.* The analysis is conducted in a combustion tube open at both ends. This is filled in the ordinary way with the substance, mixed with copper oxide or lead chromate. Through one cork of the tube passes the end of a two-way cock, the second leg of which is connected with a drying apparatus for air and oxygen, such as is ordinarily used in organic analysis. The third leg is connected with a carbon dioxide generator. The weighed calcium chloride tube passes through the cork at the

*This Journal III, 98.

other end of the combustion tube, and this in turn is connected with the apparatus for collecting nitrogen.

To conduct the analysis, the apparatus is first filled with carbon dioxide, and the nitrogen then determined in the ordinary way. When the volume of nitrogen no longer increases, the current of carbon dioxide is stopped, the nitrogen tube removed, and first oxygen and then air led through the apparatus as in an elementary analysis. The entire operation requires two to two and a half hours for its completion. Results given for urea, acetanilide, para-nitro-cinnamic aldehyde, meta-nitro-benzoic acid, brom-meta-dinitro-benzene, etc., show the method to be an accurate one.

On a New Ptomaine and on a Method of Analysis of Alkaloids. M. A. M. Delézinier. Bull. soc. chim., LI, **3**, [illegible]. The portion of this article most interesting from an analytical point of view is the description of the apparatus used to make experiments on the alkaloid in an inert gas, and in vacuo. The apparatus consists of a glass jar with openings for supplying the gas desired; for creating a vacuum; and by means of which the liquid is introduced. In the vessel there is a flat stand supporting twenty test tubes, in which the experiments are made. This stand may be made to revolve by turning a handle projecting through the top of the apparatus. The heat is applied below the tube in use by means of a platinum wire heated by a current of electricity. All the joints are made tight to prevent access of air. This very ingenious piece of apparatus needs the cut appearing in the original article for a comprehension of the construction.

C. W. W.

MINERAL ANALYSIS.

[illegible]

Eruptive Rocks from Colorado. Cross and Eakins. [illegible] [illegible] II, part 3, p. [illegible] describe and give analyses of the [illegible] rocks from Silver Cliff and Rosita, Custer Co., Col. [illegible] Rh[illegible] [illegible] Tr[illegible] [illegible] [illegible] of Peridotite [illegible] Augite [illegible] [illegible] [illegible] Analyses.

	a	*b*	*c*	*d*	*e*	*f*
SiO_2	75.20	66.03	59.78	46.03	50.47	63.49
Al_2O_3	12.96	18.49	16.86	9.27	18.73	18.40
Fe_2O_3	0.37	2.18	3.08	2.72	4.19	2.44
FeO	0.27	0.22	3.72	9.94	4.92	1.09
MnO	0.03	trace	0.14	0.40	0.11	0.16
CaO	0.29	0.96	2.96	3.53	8.82	2.30
MgO	0.12	0.39	0.69	25.04	3.48	0.66
K_2O	8.38	5.86	5.01	0.87	3.56	4.62
Na_2O	2.02	5.22	5.39	1.48	4.62	5.70
H_2O	0.58	0.85	1.58	0.64	0.58	1.04
P_2O_5	trace	0.04		0.17	0.10	trace
CO_2		trace	0.75		trace	trace
S				0.01		
TiO_2					0.51	trace
Cl					trace	
	100.22	100.24	99.96	100.10	100.09	99.90

Meteorite from Durango, Mexico.—J. E. Whitfield (Am. J. Sci., June, 1889, 439) has analyzed this meteorite with the following result:

Fe	91.48
Ni	7.92
Co	0.22
P	0.21
S	0.21
C	0.06
	100.10

Weight, 33 kilos; locality, LaBella Roca, a peak of the Sierra de San Francisco, State of Durango.

Calamine from Friedensville, Pa.—J. Eyerman (Mineralogy of Pennsylvania, Part I, 29) gives the first published analysis of this zinc silicate from the old Ueberroth mine, Friedensville, Lehigh County, Pa. Analysis:

SiO_2	24.32
Fe_2O_3	2.12
H_2O	7.86
ZnO	65.05
	99.35

Phonolite from Colorado.—An analysis of this eruptive from El Paso County, Col., by L. G. Eakins (Proc. Col. Sci. Soc. Vol. II, part 3, 169).

SiO_2	60.22
Al_2O_3	20.98
Fe_2O_3	2.21
FeO	0.51
MnO	trace
CaO	1.18
MgO	trace
K_2O	5.72
Na_2O	8.83
Cl	trace
P_2O_5	trace
H_2O	0.70
	100.15

Sp. gr. (13° C.)=2.576.

Mazapilite, a New Mineral.—G. A. Koenig (Proc. Ac. Nat. Sci., 1889, p. 45) announces the discovery and describes this new species from the Jesus Maria mine, District of Mazapil, Zacatecas, Mexico.

Color, black; streak, ochre-yellow; lustre, sub-metallic; orthorhombic. Analysis:

As_2O_5	43.60
Sb_2O_5	0.25
P_2O_5	0.14
Fe_2O_3	30.53
CaO	14.82
H_2O	9.83
	99.17

Gahnite and Columbite from Pennsylvania.—F. A. Genth (Proc. Ac. Nat. Sci., 1889, p. 50) has analyzed these two minerals from Delaware County, Pa.

Gahnite from Smedley's Quarry.

Sp. gr. 4.587

Al_2O_3	57.22
ZnO	38.14
CuO	0.06
MnO	0.70
FeO	3.55
MgO	0.26
	99.93

Columbite from Mineral Hill.

Sp. gr. 5.262—5.259.

Ta_2O_5	0.83
Cb_2O_5	76.26
SnO_2	0.16
ZrO_2	0.67
WO_3 ?	trace
VO_3	0.18
Ce_2O_3, etc.	0.34
Y_2O_3, etc.	1.78
FeO	7.65
MnO	11.29
MgO	0.07
CaO	0.66
Ignition	0.33
	100.22

NOTES.

—M. Carey Lea has discovered three new allotropic forms of silver. The first, *A*, is formed by precipitating a solution of silver nitrate with ferrous citrate. The precipitate is washed with solution of ammonium nitrate and alcohol and contains 97.23% silver, the remainder being citric acid and iron. This form is *soluble in water*. The second form, *B*, is derived from *A* by adding to the solution magnesium, cupric or ferrous sulphate, etc. This form is insoluble in water, but may be converted into the soluble form. The third form, *C*, is produced by acting on silver tartrate with ferrous tartrate. It is also insoluble, and has a golden color. When either form is spread over paper or glass, continuous brilliant films are produced. The specific gravity is less than that of ordinary silver, into which they are readily con-

verted by the action of dilute acids (Am. J. Sci., June and July, 1889).

—*Method for Cleaning Old Photographic Negatives.*—In taking photographs of objects where only one or two prints are required, the negative is often laid aside or thrown away as useless. These plates when cleaned may be used to advantage about the laboratory in various ways, but the operation of cleaning them is troublesome, requiring perhaps five or six hours' soaking in water, and then scraping off the film, which sometimes still remains hard and sticks to the plate. This may be overcome by putting the plate into a solution made by dissolving 4 oz. potassium dichromate in 8 oz. sulphuric acid and 6 oz. water. The plates are now allowed to remain five minutes, taken out, and washed with water. A solution of the above proportions will clean five dozen 3¼x4¼ plates.—FRANK R. BENNETT.

PERSONALS.

—Dr. J. U. Nef, of Purdue University, Lafayette, Ind., has been appointed Assistant Professor of Organic Chemistry at the new Clark University, Worcester, Mass.

—Mr. Albert H. Welles has been appointed Chemist at Port Kennedy Furnace, Pa.

—Dr. Paul C. Freer, sometime at Tufts College, and previously a contributor to the chemical journals with W. H. Perkin, Jr., has been appointed to the chair of General Chemistry at the University of Michigan. At the same institution, Prof. John W. Langley retains a non-resident lectureship on the metallurgy of steel.

—Mr. A. Van Zwaluwenburg has taken the place of chemist for the Vulcan Iron Works, South St. Louis, Mo.

—Mr. David H. Browne has been appointed Instructor in Quantitative Analysis at the University of Michigan.

OBITUARY.

—Dr. Percy, the eminent chemist and metallurgist, died Wednesday, June 19. He was born at Nottingham in 1817, and graduate in medicine at the University of Edinburgh at the age of 21. It was while he was physician to the Queen's Hospital in

Birmingham that he first undertook the study of the chemistry of metallurgy, and so successful was he in his purpose that in 1851, when the Government School of Mines was organized, he was appointed to the Chair of Metallurgy at the recommendation of De la Beche. For twenty-eight years he held this position and contributed in a large measure to the success of this Institution, resigning his office in 1879. He was also superintendent of the ventilating arrangements in the Houses of Parliament. His works on metallurgy are standard authorities (Chem. News **60**, 11). A. H. W.

NEW BOOKS.

INORGANIC CHEMISTRY. BY IRA REMSEN. NEW YORK: HENRY HOLT & CO., 8 VO., 827 PAGES.—This book belongs to the so-called "American Science Series—Advanced Course," and is evidently intended for those who already have some knowledge of the subject. The experiments illustrating the statements advanced have been gathered into an appendix, and this certainly is a wise arrangement in a book of this kind. Prof. Remsen has evidently tried so far as possible to treat the elements in the "periodic" order, but he finds himself much embarrassed by the difficulty of doing so and at the same time giving the subject an orderly arrangement. This has led to many inevitable departures from the original plan, and it is, we think, almost to be regretted that the attempt was made. Chemists, like other people, must follow the fashions, and it is quite possible that the sale of a text-book which was not "periodic" might suffer. In saying this we do not mean to minimize in the least the work of Newlands, Mendelejeff and L. Meyer; but new ideas are oftentimes too hard worked, and we think this is the case when the periodic arrangement is used in a text book.

It may as well be acknowledged at once that the periodic law in its present form accounts for only a part of the self-evident relationships between the elements; and Prof. Remsen acknowledges this in substance on page 159, when he says, "Thus, as will be seen in due time, the elements aluminum, chromium, manganese, and iron are analogous in some respects, but by following the plan sketched they will be taken up in different groups." Oxygen

is first treated, then hydrogen, chlorine, bromine, iodine, fluorine, manganese, sulphur, selenium, tellurium, nitrogen, etc., in the order named. The metallic elements are, for the most part, treated together, beginning with the alkali metals, then follow the alkaline-earth metals, etc.

The theoretical side of the subject claims a large share of attention throughout the book, and is treated clearly and in the author's usual common sense fashion. The views stated are those which the facts seem to warrant, as, for example, on page 428, the author states that "Valence, as we have seen, is not a constant property of the atoms." It is not too common to find chemists who are willing to acknowledge this self-evident fact.

Prof. Remsen certainly did wisely in writing this book. Only a few of the books written nowadays have any excuse for existence, but this is one of the few. E. H.

AN INTRODUCTION TO THE STUDY OF QUALITATIVE CHEMICAL ANALYSIS. BY J. T. MCGILL, PH. D., ADJUNCT PROFESSOR OF CHEMISTRY IN VANDERBILT UNIVERSITY, NASHVILLE, TENN. PRINTED FOR THE AUTHOR. 12 MO., 48 PP. MAILING PRICE, FORTY CENTS. 30 pages are consumed before Qualitative Analysis proper is reached, in treating of length, volume, weight, the Bunsen burner, temperature of ignition, the blowpipe and blast-lamp, charcoal as a reducing agent, etc. A short extract from the section on qualitative analysis will perhaps give a good idea of the method of treatment.

"**SUB-GROUP B.**

Sulphides Insoluble in Ammonium Sulphides. (*a*) Which members of Sub-group B are precipitated from solutions by H_2SO_4? by NH_4OH? Which sulphides of Group II. B are dissolved by boiling with HNO_3? by boiling with H_2SO_4? by KCN solution? Make out the results of these experiments in tabular form. From the above reactions make out a scheme for the separation of the members of Sub-group B."

When we need a new coat and have no time to make it, we select one from the stock on hand, pay for it and wear it. If you need a new book on Qualitative Analysis, buy a copy of this book and try it on. If it fits you, get some more and use them. E. H.

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NOTES ON THE BECHI TEST

FOR COTTON-SEED OIL IN LARD.

BY D. WESSON.

The Bechi test being regarded by most of the best authorities as one of the best, if not the best, single qualitative test for the presence of cotton-seed oil in lard, we decided to make some experiments with pure lard from various sources to see what effect nitrate of silver would have on the same, and what influence certain peculiarities in the character of the lard would have on the results.

2 gms. of nitrate of silver were dissolved in 200 cc. 95 per cent. alcohol, 40 gms. of ether added, and the whole shaken together. The solution was exposed to sunlight till precipitation ceased, and was then carefully decanted into a bottle of dark glass to protect it from any influence of light.

The test was applied by adding 5 cc. of the solution to 10 gms. of the melted lard in a 60 cc. cylindrical bottle, such as is commonly used for oil or syrup samples. After shaking the lard and solution well together, the bottle is placed either in a steam or water bath and heated, with occasional shaking for fifteen minutes, at the end of which time the greater part of the alcohol and ether had been driven off and the reaction with nitrate of silver had taken place. With pure cotton-seed oil, or where large quantities of cotton-seed oil were present, a mirror of metallic silver was deposited and the oil colored a deep greenish hue. Small amounts gave a deep red coloration, with more or less deposit of metallic silver. Lard either darkened slightly or gave a purplish coloration, with little if any reduction of metallic silver.

Date.	Sample No.	Packer.	Delivered in.	Quality.	Free Acid. Per Cent.	Action with Nitrate of Silver.
April 13	1	X	. . .	Good	. .	Pink, slight reduction.
" 13	2	X	. . .	"	. .	Light pink, slight reduction.
" 13	3	X	. . .	"	. .	Pink, slight reduction.
" 15	4	D	Tank Car	Choice	.65	Blackens, slight reduction.
" 15	5	A	Tank Wagon	"	.45	Reddens, no metallic silver.
" 16	6	B	Tank Car	Fair	.70	Reddens and darkens, slight reduction.
" 16	7	A	Tank Wagon	Choice	.35	Light reddish-brown, no reduction.
" 16	8	A	"	"	.35	Light reddish-brown, no reduction.
" 16	9	A	"	"	.40	Light reddish-brown, no reduction.
" 17	10	A	"	"	.35	Light reddish-brown, no reduction.
" 18	1	A	"	"	.45	Light reddish-brown, no reduction.
" 18	2	A	"	"	.35	Light reddish-brown, no reduction.
" 18	13	D	Tank Car	Fair	.70	Blackens a little and reddens, slight reduction.
" 19	14	D	"	"	.50	Reddens a little, no reduction.
" 19	15	D	"	"	.65	Darkens some, no reduction.
" 19	16	A	Tank Wagon	Choice	.35	Reddens, no silver.
" 19	17	A	"	"	.40	Reddens a little, no silver.
" 19	18	D	Tank Car	Dark	.75	Blackens. Rise of temperature with H_2SO_4 1° over standard.
" 19	19	C	Tierces	Fair	.40	Reddens, no silver.
" 22	20	B	"	"	.50	Reddens and darkens slightly, no silver.
" 22	21	A	Tank Wagon	Choice	.35	Reddens and darkens slightly, no silver.
" 22	22	A	"	"	.35	Reddens and darkens slightly, no silver.
" 22	23	A	"	"	.40	Reddens and darkens slightly, no silver.
" 23	24	A	"	"	.40	Reddish brown.
" 23	25	A	"	"	.40	Reddish brown.
" 24	26	E	Tierces	Fair	. .	Darkens, slight reduction.

In most cases the color was quite different from that produced by cotton-oil, though in some instances results were somewhat doubtful.

The samples of lard tested were from different lots shipped by different packers, and the quality in most cases was "choice." The best lard, as a rule, showed the lowest percentage of free fatty acid. The results are shown in the table.

Referring to the table, the first column shows the date the shipment was received and the test made; the second column the number of the sample. The third column designates the packer.

A. is a Chicago packer of first-class reputation, the quality of whose lard has always been very choice and of unquestionable purity.

B. is another local packing concern, who we have every reason to believe never saw a barrel of cotton oil on their premises. The same can be said of C., whose lard is standard quality, and as a rule choice.

D. is an Omaha concern, and E. is from Cedar Rapids, Ia.

Samples 1, 2, 3, marked X, were taken from cellar barrels, and the makers are not known.

The fourth column shows manner of delivery, and also quantity represented by sample, when it is considered that a tank car holds about 33,000 lbs., a tank wagon 10,000 lbs., and tierces are delivered in lots of from fifty up to two hundred and fifty.

The fifth column shows the quality of the lard as judged from a commercial standpoint, color and flavor being the chief considerations.

The sixth column shows the free acid of the sample. This is one of the most important characteristics of a lard, inasmuch as it is an index of the care used in preparation.

Other things being equal, the lower the free acid the better the lard.

Prime lard often runs as high as one or one-and-a-quarter per cent. of free acid, but choice lard seldom exceeds 0.75 per cent. The amount of acid present is influenced by two things: first, the time the fat remains unrendered, and second, the temperature at which it is rendered, combined with length of the rendering process. In either case the splitting up of a portion of the glycerides is evi-

dence that the fat has undergone change, and we should naturally expect some different reactions with so sensitive a reagent as nitrate of silver. This appears to be the case, and to these differences in handling of the fat and rendering we ascribe the different results obtained in our investigations.

As an example of the effect of handling on fat, we recently obtained some pieces of caul and kidney fat from some cattle that have been fattened on cotton-seed meal. For some reason we were unable to render the samples immediately, and they were allowed to stand several days in a cool place, when the fat was carefully rendered in the laboratory and filtered. With the nitrate of silver test a deep red coloration was produced indicating about ten per cent. of cotton-seed oil. We tried the Millian modification and found the fatty acids did not react in the slightest. Our samples showed 5 per cent. of free acid. At the same time we tested a sample of "butter-stock" made from the cauls and kidneys of the same cattle, and found it gave absolutely no reaction.

Referring to the table, sample No. 18 appeared doubtful to us, and we found that with sulphuric acid it gave a rise of one degree over standard lard. This would have indicated, leaving other facts out of consideration, about 2½ per cent. of cotton-oil. As a matter of fact, 2½ per cent. of cotton-oil would not have caused so much blackening, and we have to account for the same in another way. The flavor and color of the lard showed that it had been probably cooked in a high temperature, and the per cent. of free acid present showed that it had not been rendered any too promptly. We have seen such lard often, and it always gives a higher rise of temperature with sulphuric acid than the average run.

We find that cotton-seed oil which has been heated till it begins to smoke for a minute or two loses its power of reducing silver nitrate, and the same is true of oil which has had a current of air passed through it for several days.

We conclude from our experiments that coloration with nitrate of silver is no indication of the presence of cotton-seed oil unless actual metallic silver is deposited. Almost any pure commercial lard will give more or less color, varying with the age of the sample and care used in rendering. In applying the test, the

character of the lard must always be taken into consideration, and in no case, except in fresh samples, when cotton oil is present in quantities over ten per cent., should the evidence be taken as conclusive. As an indication of small amounts of cotton-oil in old samples or stock rendered under certain conditions, we believe the test to be without value.

CHICAGO, August 7, 1889.

THE EXAMINATION OF OLIVE OIL FOR ADULTERATION.*

BY THOS. B. STILLMAN, PH. D.

This subject is one of considerable importance, not only from a chemical, but also from a sanitary standpoint. Any sophistication practiced must be of such a nature as not to be of easy identification.

Tests used as standard ones for the examination of olive oil a few years since now utterly fail to indicate the adulteration.

Especially so is this true of the former tests used for distinguishing cotton-seed oil when mixed with olive oil.

Even some of the tests given in the late edition of the U. S. Dispensatory for the identification of a pure olive oil are entirely misleading and valueless.

No one test has been found sufficient for this purpose (as in the case in laboratories of some State Inspectors), but by a careful comparison of the following five tests any adulteration of an olive oil should be indicated, also the nature of the oil added: (1), Specific Gravity; (2), Rise of Temperature when the oil is treated with concentrated sulphuric acid; (3), the Elaïdin Test; (4), Iodine Absorption, and (5) Nitric Acid Test.

Two of these tests, the elaïdin test and the iodine absorption require considerable chemical experience for their proper treatment, and it will be found that the subject is not always one of simplicity and ease.

1. *The Specific Gravity.*—Pure olive oil varies between .915 and .917, any adulteration with heavier oils such as cotton-seed (specific gravity .926), poppy-seed (specific gravity .927), sesamè (specific gravity .924), will be indicated. An addition of 5 per cent. of

*From the Stevens Indicator.

any of these oils might not be indicated absolutely, but I have never found an olive oil when adulterated to contain less than 20 per cent. of the adulterant; any less amount would not pay for the trouble of handling and mixing the different oils.

Twenty per cent. of any of the above oils is easily shown by this test, and the identification between these oils is left to other special tests.

Lard oil (specific gravity .915), and tallow oil (specific gravity .916), sometimes used for the adulteration of olive oil, would not be indicated by this test.

The oils most largely used for adulterating olive oils for the American market are lard oil and cotton-seed oil.

The specific gravity test will readily indicate if any cotton-seed oil has been added, but the lard oil must be identified by other means.

2. *Rise of Temperature with Sulphuric Acid.*—This test is performed as follows: 50 cc. of the olive oil are placed in a beaker and 10 cc. of strong sulphuric acid added slowly with constant stirring. During this the temperature of the liquid is carefully noted by a thermometer.

Olive oil increases in temperature 41°–43° C., cotton-seed increases 70°–75° C., hemp seed 98° C., sesamè oil 68° C., lard oil 42° C., corn oil 43° C. This is one of the best tests for the presence of cotton-seed oil; not only is the increase of temperature very marked (nearly twice as great as for olive oil), but the action of the sulphuric acid is characteristic, violent ebullition often forcing the liquid out of the beaker.

Five per cent. of cotton-seed oil in olive oil can be detected by this test, provided no other seed oil is present.

This is easily proven synthetically by taking a sample of pure olive oil and adding thereto exactly five per cent. of cotton-seed oil, making the test and noticing the rise of temperature.

The difference between the figures obtained and that required for olive oil will be found to indicate five per cent. of cotton-seed oil. Lard and tallow oils cannot be identified in olive oil by this test.

3. *The Elaïdin Test.*—This test is made as follows: (Allen Organic Analysis, page 57) "1 cc. of mercury should be dissolved

in 12 cc. of cold nitric acid of 1.42 specific gravity. 2 cc. of the freshly-made *deep green* solution is then shaken in a wide-mouthed stoppered bottle with 50 cc. of the oil to be tested, and the agitation repeated every ten minutes during two hours. When treated in this manner, oils consisting of olein approximately pure, or of mixtures of olein with the solid glycerides, give a solid product of greater or less consistency. Olive oil is remarkable for the canary or lemon yellow color and great firmness of the elaïdin yielded by it.''

Generally speaking, this test is a good one for the identification of olive oil when mixed with seed oils.

The mass becomes solid if the oil is pure olive within from 2 to 10 hours, whereas, if cotton-seed oil be present, the mixture remains liquid and does not solidify at all.

It is generally stated that two hours' exposure of the mixture is sufficient for the solidification of a pure olive oil.

I have not found this to be the rule, but rather the exception.

An olive oil pure and *fresh* will fulfill these conditions, but one that has been exposed to sunlight or is not fresh requires from four to ten hours for solidification. This test is highly recommended in the writings of chemists who have made a specialty of the examination of olive oils, but since lard oil (a rather recent product) has been used as an adulterant, the test has not the prominence of former years.

Lard oil as well as tallow oil react in a similar manner to olive oil in this test, whereas all other oils can probably be detected. No doubt must exist in the operator's mind concerning the condition of the oil after the test is made. If the resulting mass is not *hard* and *solid*, then other oils than olive are present.

4. *Iodine Absorption.*—This test, also called Hübl's (Ding. Polyt. Journal, ccliii, 281), is a very valuable one in the identification of olive oil when the latter is adulterated with any of the following oils: earth-nut, rape-seed, cotton-seed, poppy-seed, sesamè.

Pure olive oil indicates 81–84; cotton-seed oil, 105–108; rape-seed oil, 100–105; earth-nut, 101–105; sesamè, 105–108. It is seen that an admixture of olive oil with any of the above seed

oils can readily be detected. It fails, however, with lard oil, as the latter has an index of 82.

Any fat or oil containing much stearic acid will give a low iodine absorption, as the stearic acid does not combine with the iodine, whereas, the oleic acid does. Thus lard gives a low absorption figure, 59, but when the lard oil is pressed from the lard (separated from the stearine) it becomes principally oleic acid and the iodine absorption rises to 82.

5. *Color reaction with Nitric Acid.*—There are many different methods of applying this test, but the following, called Massie's test, gives good results, viz.:

Agitate three parts of the oil with one part of nitric acid (sp. gr. 1.40) for two minutes, in a small test tube. The resulting colors after separation of the two fluids give an idea of the oil present.

Pure olive oil of first pressing will not change in its color upon the addition of nitric acid, or, at most, becomes yellowish-green. The addition of any seed oils is at once evident by the change in color, cotton-seed oil showing a bright red color turning to brown. This change is immediate.

Lard oil, as well as tallow and corn oil, show a reddish brown coloration.

This nitric acid test will indicate the adulteration of the above oils with pure olive oil of the first pressing, but many brands of pure olive oil are upon the market that are not composed of the first pressings of the olive only, but of a mixture of the first with the second, and even third, pressings. The latter two are pure olive oils, but their reactions are entirely different as regards the nitric acid and elaïdin test, and might indicate to an inexperienced chemist the adulteration with lard oil or even cotton-seed oil.

The second and third pressing of the olive produce an oil containing more of the gum and vegetable albuminous principles of the olive than the first pressing, and such an oil reacts with a yellowish red color when treated with nitric acid; the elaïdin test is also retarded. The amount of pure olive oil, of first pressing only, is small in this market, whereas, the amount produced by mixing the different pressings, is very large.

This, I think, accounts in a measure for the fallacy that pure olive oil is almost unknown in the American market.

The usual color test for cotton-seed oil, somewhat modified, would be given by an olive oil of second and third pressings. Even Bechi's silver test, which is considered a good test for the presence of cotton-seed oil in olive oil, reacts the same on an olive oil composed of the different pressings as for pure cotton-seed oil. However, the specific gravity test, the rise of temperature with sulphuric acid, and the iodine absorption test are the same with an olive oil of different pressings as with an oil of first pressing, and by these tests the purity is determined. The U. S. Dispensatory, page 1026, gives the following test, among others, for the identification of olive oil:

"If one gramme of oil be agitated in a test-tube with two grammes of a cold mixture prepared from equal volumes of strong sulphuric acid and nitric acid of specific gravity 1.185, and the mixture set aside for half an hour, the supernatant oily layer should not have a darker tint than yellowish." And

"Nor should a green or red layer separate on standing if one gramme of the oil be shaken for a few minutes with one gramme of a cold mixture of sulphuric acid (specific gravity 1.830) and nitric acid (specific gravity 1.250) and one gramme of disulphide of carbon."

Attention is drawn to these two tests taken from the U. S. Dispensatory, as they were the basis of the condemning by a New Jersey State official of pure olive oil.

A few months since an olive oil was introduced into this market from Aix. It was at once condemned as impure by the inspector, who claimed that the impurity was cotton-seed oil.

A sample submitted to me by the importer failed to show any adulteration.

This was not satisfactory, however, to the inspector, who demanded a referee. One was chosen, who, upon chemical examination of the oil, pronounced it pure olive oil.

Still the official was not satisfied, claiming that the oil did not fulfill the tests required by the U. S. Dispensatory, citing the two tests above quoted.

If such tests be recognized as official, then the following well-

known brands of pure oil should also be condemned: Barton & Guestier, Bordeaux; Porter & Co., Tuscany; Alexis Godillot, Jeune, Aix; Massereau & Bareil, Aix; Gordon & Dilworth (Saint Bonnet), New York.

These oils have been tested by me with the results that they are pure oils, but not a single one complies with the tests given in the U. S. Dispensatory, each and every one giving either an orange red or a red color, instead of an unchanged yellow. A test somewhat similar to this, which has been in use in my laboratory, rarely fails to show the presence of lard oil or tallow oil, also corn oil, in olive oil. It is as follows: Agitate in a test-tube 3 parts of the oil under examination with 3 parts of chloroform, then add 2 parts of an equal mixture of strong nitric and sulphuric acids. Repeat the agitation a few times for ten minutes, then set aside for an hour.

Olive oil composed of the different pressings will react the same as of the first pressing, maintaining a faint yellowish green color,—and it is the only color test I know of in which the reactions are alike—for these two different classes of olive oil. When lard oil or tallow oil are present they produce a brownish red coloration which is very characteristic.

Cotton seed oil produces a rich red color, and corn oil a deep lake color.

If any sample of olive oil under examination gives a specific gravity of .914–.918, a rise of temperature of 40° C.–43° C. fulfills the conditions of the elaïdin text for olive oil, gives an iodine absorption of about 82, and a brownish red color with the acid and chloroform test, lard oil is present as an adulterant of the olive oil.

The reactions of lard oil are nearly all identical with olive oil, and oftentimes it is exceedingly difficult to distinguish the presence of the former; but by a careful comparison of the first five tests given, and with the chloroform and acid test, no doubt should exist.

The following determinations made by Mr. D. A. Outcalt, in my laboratory, show the results obtained upon a few samples of oils:

Specific Gravity.

Barton & Guestier, olive oil	0.9160
Porter & Co. "	0.9165
Alexis Godillot, Jeune "	0.9170
Massereau & Bariel, "	0.9170
Gordon & Dilworth, "	0.9165
Lard oil	0.9165
Corn oil	0.9222
Cotton seed oil—	0.9262
Societe Hygienique Alimentaire (olive oil)	0.9192

Rise of Temperature with H_2SO_4.

Barton & Guestier, Olive oil	40.5° C.
Porter & Co. "	43° C.
Alexis Godillot, Jeune "	41° C.
Massereau & Bariel "	42° C.
Gordon & Dilworth "	42° C.
Lard oil "	42° C.
Corn oil "	43° C.
Cotton-seed oil "	75° C.
Societe Hygienique Alimentaire	66° C.

Elaïdin Test.

Elaïdin Test.	Iodine Absorption.	Nitric Acid Test.	Acid Test from U. S. Dispensatory.	Chloroform and Acid Test.
Barton & Guestier, 6 hrs.	81.2	Yellow at first, finally faint red	Orange red	Yellow
Porter & Co., 6 hrs.	79.4	Yellow	Green, then red	Yellow
Alexis Godillot, Jeune, 5 hrs.	79.8	Yellowish green at first, then reddish	Orange red	Yellow
Massereau & Bariel, 5 hrs.	79.	Yellowish at first, then reddish	Reddish	Yellow
Gordon & Dilworth, 6 hrs.	81.	Yellowish at first, then reddish	Orange red	Yellow
Lard oil, 5 hrs.	83.	Yellow, then faint red	Red	Brown-red
Corn oil, did not solidify	88.9	Brick red	Dark red	Deep lake
Cotton-seed oil, did not solidify	120.4	Red	Red	Red
Societe Hygienique Alimentaire (olive oil) did not solidify	107.3	Red	Red	Red

An examination of these results shows that the first five oils are pure olive oils, and that the one marked and sold under the name "Société Hygienique Alimentaire," as a pure olive oil, contains a large amount of cotton-seed oil. The reactions of lard oil, corn oil, and cotton-seed oil are added for comparison.

A BOTTLE FOR STORING AND TRANSPORTING HYDROFLUORIC ACID.

BY EDWARD HART.

Hydrofluoric acid is in comparatively little use by analysts because of the trouble involved in making it and the expense of the necessary platinum bottle for keeping it. Most analysts are entirely too busy to make their own chemicals, and hence they learn to do without this very convenient reagent.

After a good deal of study and work, I have devised the following form of bottle, which answers the purpose perfectly and is inexpensive. The bottle is made either of paraffine, wax or ceresine. In most instances ceresine is preferable, since it melts at a higher temperature. Neither of these substances is attacked by hydrofluoric acid, which keeps indefinitely in them without change.

The bottle is shown in Figs. 1 and 2. Fig. 1 shows the bottle with ceresine stopper in position for use. In Fig. 2 the stopper is inverted and sealed to the rim of the bottle for transportation. When received by the consumer, a knife is inserted at *a*, and the stopper cut loose and inverted. The pure acid can now be obtained of Messrs. Baker & Adamson, of Easton, Pa., put up in 4 oz. bottles. These are securely sealed, and packed in sawdust in tin cans for shipping. Vessels of various sizes and shapes for containing hydrofluoric acid and for experimenting with it, such as funnels, dishes, test-tubes, etc., may readily be made of paraffine, wax, or ceresine, and will often be found very convenient.

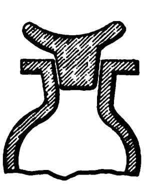


Fig. 1.

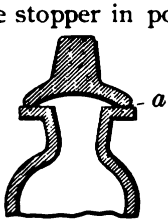


Fig. 2.

In looking over the books, I have been unable to find any specific gravity tables for hydrofluoric acid, and have therefore

constructed a rough table for my own use, which has been convenient. It rests upon the two determinations marked below with an asterisk, and is probably accurate to within one per cent.

Approximate Table for Hydrofluoric Acid. 15° C.

Sp. gr.	Per cent. H. F.	Sp. gr.	Per cent. H. F.	Sp. gr.	Per cent. H. F.
1.01	2.90	*1.10	29.00	1.19	55.10
1.02	5.80	1.11	31.90	*1.20	58.00
1.03	8.70	1.12	34.80	1.21	60.90
1.04	11.60	1.13	37.70	1.22	63.80
1.05	14.50	1.14	40.60	1.23	66.70
1.06	17.40	1.15	43.50	1.24	69.60
1.07	20.30	1.16	46.40	1.25	72.50
1.08	23.20	1-17	49.30		
1.09	26.10	1.18	52.20		

ESTIMATION OF BROMINE IN PRESENCE OF CHLORINE.

BY A. B. PRESCOTT AND W. L. DUNN.

For estimation of bromides taken in mixture with chlorides indirect methods of analysis have been, until within about ten years, almost the only trustworthy dependence. The reactions used for actual quantitative separation did not go further than removal of an excess of the chlorides to obtain a concentration of the bromides preparatory to their final estimation. In late years, however, trial has been made of certain reactions for complete separation, in methods of direct analysis, and it can no longer be said that every direct method is unsafe. Seeking, first, the best of the known methods for indirect estimation, and then the best of the proposed methods for direct estimation, the writers have undertaken a set of control analyses with each method so adopted, and the results are given below.

I. For an *indirect estimation*, starting with a weighed quantity of silver bromide and chloride, as the one factor, it was decided

to take metallic silver as the other required factor. The reduction to metallic silver was adopted, after collating the authorities, as capable of closer results than those obtained by conversion of the mixed bromide and chloride to entire chloride of silver according to Wittstein* and others; or conversion to entire bromide of silver by digestion with potassium bromide solution according to Field,† Siewert,‡ or Huschke,§ these methods providing for estimation of iodide as well. The conversion first to bromide and then to the iodide, by intervention of cyanide solution, after Maxwell Lyte,‖ though favorably reported upon by Whitfield¶, is certainly more complex than conversion to metallic silver. The reduction of the silver haloids by heating in a stream of hydrogen, according to the earlier directions of Fresenius, was never wholly satisfactory and may well be superseded by electrolytic reduction. At an early period the electrolysis of the fused mass was proposed by Bolley.** Careful control analyses were made, a few years ago, by Kinnicutt,†† who applied the poles to the fused mass covered with dilute sulphuric acid. A more favorable condition for the electrolysis is that of the cyanide solution of the silver haloids,‡‡ a solution obtained directly from the argentic precipitate, whereby fusion and its disadvantages are avoided. Mr. Whitfield of the U. S. Geological Survey has recently subjected the plan to a careful trial and with his report§§ has given a good account of methods hitherto in use. He found good results by the deposition of metallic silver from the cyanide solution; also good results by the precipitation of silver iodide from the same solution. And the directions given by Whitfield were taken as an outline of the operation for indirect

[illegible]

analyses, in this work, the operation being finally adjusted as follows:

Of the mixed chloride and bromide, 5 gms. were taken. The complete silver precipitate, obtained as required in gravimetric work, and carefully protected from the light, was filtered through a weighed asbestos filter. The filter found best for this purpose is prepared as follows: A carbon-tube* is selected, a light plug of absorbent cotton is placed in the bottom, and then a layer of very thoroughly washed asbestos about one-fourth of an inch in thickness. The filter was dried, to a constant weight, in an air-bath at 150° C., cooling in a desiccator. The precipitate well washed on this filter was dried at the same temperature in the same bath and weighed under equal conditions, to obtain the weight of the silver chloride and silver bromide. The precipitate is then dissolved on the filter by application of potassium cyanide solution, using the least possible quantity of the solvent, while completely washing out the solution into a weighed platinum dish, which is in contact with the negative pole of a battery of two Bunsen cells. The positive electrode is introduced into the solution, but not brought in contact with the dish. The current is continued until the solution is destitute of silver, as shown by a drop of the solution taken upon a glass slide, over a dark ground, remaining entirely clear while treated with a drop of hydrochloric acid. As soon as the silver is completely deposited, the dish is removed from the battery, drained, and several times washed with water, to prevent the solvent action of the cyanide upon the metallic silver. The dish with the silver deposit is dried in the air-bath, cooled, and weighed. For both weighings of the dish, its temperature is equalized in the balance-case.

The results of *seven* control analyses by this method, in the indirect way, are given further on.

II. For a *direct estimation*, the separation of the bromine by the action of chromic acid, in distillation, was adopted. The reactions of chromic acid with chlorides, bromides, and iodides, under specific conditions, have been reported upon† for many

*In the form of a test-tube of full width, perforated on the bottom, where a section of glass tubing is fused on, over the perforations, serving as a funnel-neck.

†WOHLER, 1834: *Ann. Phys. Chem. Pogg.* **33**, 343. DONATH, 1880: *Zeitsch. anal. Chem.*, **19**, 20.

years. Adaptation to the quantitative separation of the haloids was made by Dechan in 1886*, who sets forth, what was partly shown by Donath, that having dichromate in proper concentration, on boiling, iodine is liberated from iodides and distilled over in complete separation from bromine. Then, on adding sulphuric acid in due quantity, and maintaining a certain concentration, bromine is all distilled over, while chlorine is left behind, greater concentration being required to distil any chloro-chromic anhydride. As finally adjusted in this experimentation, the process is as follows:

A distilling flask of fully 150 cc. capacity admits a separatory funnel, closed with a stop-cock, the funnel-tube reaching the bottom of the flask. The vapor-tube of the distilling flask, bent just above the stopper to a line slightly descending for four or five inches, and then turned down vertically, forms the inner tube of an upright Liebig's condenser of about eight inches length, when it passes to the bottom of a receiving flask of about 100 cc. capacity, and with twice tubulated stopper, the second tubule connecting with an aspirator. The distilling flask is adjusted on a retort-stand over a small Bunsen burner directly applied. Of potassium dichromate 50 gms. are placed in the distilling flask, and 100 cc. of distilled water are added, with 0.5 to 1.0 gm. of the haloids, the material in which the bromine is to be estimated. With very low percentages of bromine there may be taken 2.0 grams of the material. The distilling flask being in position, connected with the vapor tube and the receiving flask, heat is applied until the dichromate is all dissolved. If iodine be present, the distilling liquid is boiled, with the aspirator in action and the funnel-tube open, until the distillate ceases to turn starch water blue, when, if desired, the iodine can be estimated as directed below for bromine. The receiving flask is now cleaned and charged with 10 cc. of thirtieth normal solution of arsenious acid; the distilling flask is charged with 10 cc. of sulphuric acid previously diluted to 20 cc.; and the distilling liquid is boiled, while the aspirator is in action, the funnel-tube open, and the condenser kept cold. The contents of the distilling flask are maintained at a volume of 100 to 120 cc. After boiling about an hour, the flame is removed, the receiving flask detached, with rinsing of

*M. Dechan, 1886: *Jour. Chem. Soc.* (Trans.), **49**, 682.

the delivery tube, a test-tube containing some drops of potassium iodide solution and starch water placed as a receiver, and the flame replaced until the distillate is tested for absence of bromine. When the distillation is complete, the contents of the receiving flask are titrated with thirtieth normal solution of iodine, this solution having been compared with that of arsenious acid. The quantity of iodine used is calculated into its equivalent of bromine.

Other methods of direct estimation depending upon the action of oxidizing agents other than chromic acid, upon the graded differentiation of the haloids, are methods* with permanganate, manganese dioxide, and hydrogen peroxide. Such a partial separation of the haloids as has been found serviceable for concentration of bromine, is effected by fractional precipitation with the silver, by solubility of alkali salts in alcohol,† and by the manganese dioxide as above.‡

I. By Indirect Method.

	Na Cl taken.	KBr taken.	Br taken.	Br found.	Error.	Error per 1 of material.
1	5.000	0.020	0.01343	0.013320	0.000110	0.000022
2	5.000	0.010	0.006715	0.006500	0.000215	0.000043
3	5.000	0.010	0.006715	0.005920	0.000795	0.000156
4	5.000	0.0082	0.005506	0.005405	0.000101	0.000020
5	5.000	0.0062	0.004163	0.003020	0.001143	0.000228
6	5.000	0.0100	0.006715	0.006620	0.000095	0.000019

*MOHR, manganese dioxide, 1855: *Ann. Chem. Phar.*, **93**, 80.
VORTMANN, manganese and lead dioxides, 1880: *Ber. d. chem. Gess.*, **19**, 352.
BERGLUND, Vortmann's method, 1885: *Zeitsch. anal. Chem.*, **24**, 196.
BERGLUND, use of permanganate, 1885. *Zeitsch. anal. Chem.*, **24**, 184.
E. HART, permanganate in qual. anal., 1885: *Zeitsch. anal. Chem.*, **24**, 182.
CAZAZZI, on barium dioxide, 1886: *Gaz. Chim. ital.*, **18**, 174.
COOK, use of hydrogen peroxide, 1885: *Jour. Chem. Soc.*, **47**, 462.
SCHOENE, the same, 1879: *Ann. Chem. Phar.*, **195**, 228.

†FEHLING, 1848: *Jour. prakt. Chem.*, **45**, 269.
‡MARCHAND, 1849: *Jour. prakt. Chem.*, **47**, 363.

II. By Direct Method.

	Na Cl taken.	KBr taken.	Br taken.	Br found.	Error.	Error per 1 of material.
1	0.480	0.020	0.013431	0.013559	0.000128	0.000256
2	0.480*	0.020	0.013431	0.012862	0.000569	0.001138
3	0.563	0.010	0.006715	0.005583	0.001132	0.001971
4	0.483	0.010	0.006715	0.006431	0.000284	0.000573
5	0.400†	0.010	0.006715	0.006640	0.000075	0.000183
6	0.485	0.015	0.010072	0.009910	0.000162	0.000324
7	0.490	0.010	0.006715	0.006581	0.000134	0.000268

In the first four experiments by the direct method, decinormal solutions of arsensious acid, and of iodine, were used. The more dilute solutions, thirtieth normal, gave better results. Taking 10 cc. of the thirtieth normal solution, as above, the quantity of material taken should be such as to carry not much above 0.013 of bromine.

UNIVERSITY OF MICHIGAN,
ANN ARBOR, August, 1889.

THE MOLECULAR WEIGHTS OF CERTAIN SUBSTANCES AS INDICATED BY THE BOILING POINTS OF THEIR SOLUTIONS.

BY H. W. WILEY.

The apparatus with which the experiments to be described below were made, was devised in August of this year, and the experiments were commenced about the first of September. It was not my intention to publish the results of this work until they had been greatly extended, but the notice of the observations of Ernest Beckmann of his employment of the same method induces me to submit the data already obtained for publication.

I have no desire whatever to question Mr. Beckmann's priority

*Of sodium chloride and mangnesium sulphate.
†Of sodium and calcium chlorides and sulphates.

in the use of the method, but it may be of interest to know that it was independently adoped by me and all the data submitted below obtained before a notice of his work reached me in the "Chemisches Central-Blatt," No. 7, 1889, this day received.

The apparatus employed consisted of an oval round bottom flask of about 200 cc. capacity, the neck of which was supplied with a side tube for the purpose of connecting it with an appropriate condenser. By this means the volume of the liquid employed was kept constant. The thermometer employed was accurately graduated to tenths, the distance between each tenth of a degree being so great that with the aid of the cathetometer used in reading it we could easily distinguish the .02 of a degree. The bulb of the thermometer was made to occupy the central portion of the liquid, the volume of which in all cases was 150 cc. To protect the bulb of the thermometer against the influence of the steam bubbles, it was covered with a double coating of fine copper foil. This method of coating the bulb of the thermometer of course precluded its use with substances easily reduced, such as silver and mercury salts.

Sodium chloride was the substance chosen to determine the factor for use. Carefully repeated determinations with this substance gave the following data :

Substance taken	Sodium chloride.
Volume of water	150 cc.
Weight of substance taken	4.5 gms.
" " " " to 100 cc. of water .	3.0 "
Boiling point of water	$99^\circ.47$
" " " sodium chloride in solution . .	$99^\circ.93$
Total rise of temperature	$^\circ.46$
Rise of temperature for 1 gm. per 100 of substance	$^\circ.1533$
Molecular weight of sodium chloride	58.5

Factor $= .1533 \times 58.5 = 8.968$.

Using this factor as a constant, the following determinations have been made :

Substance Taken.	Volume Water.	Weight Substance taken in gms.	Weight Substance taken in gm. to 100 cc. water.	Temperature of Boiling Water. C°	Boiling Temperature of Substance in Solution. C°	Total Rise of Temperature. C°	Calculated Molecular Weight	Theoretical Molecular Weight
KCl	150	6.0	4.0	99.50	99.85	.35	76.91	74.5
KBr	150	6.0	4.0	99.50	99.79	.29	123.7	119.0
KI	150	9.0	6.0	99.50	99 83	.33	163.1	166.0
KNO_3	150	6.0	4.0	99.50	99.83	.33	108.7	101.0
$K_2Cr_2O_7$ . . .	150	18.0	12.0	99.50	99.88	.38	283.2	295.0
$NaNO_3$	150	6.0	4.0	99.44	99.86	.42	85.4	85.0
Sucrose . . .	150	20.0	13.33	99.50	99.70	.20	643.2	342.0
Oxalic Acid .	150	6.0	4.0	99.50	99.70	.20	179.4	90.0

In addition to the numbers given above, a large number of determinations have been made with substances containing water of crystallization. In those cases the calculated molecular weight does not correspond either with the theoretical molecular weight, including the water of crystallization, nor with the molecular weight of the anhydrous salt. These results seem to indicate that substances containing water of crystallization exist in solution in a modified form, and the influence of this modification on the boiling point of the solution can only be determined by largely extended observations.

It is curious to note further that in the two organic substances examined, the observed molecular weight obtained, using the same factor obtained by observations on the sodium chloride, is just about double the ordinary accepted molecular weight.

Since the factor for boiling solutions is so much less than that for the freezing mixtures, the determination of the molecular weight by the above method requires a solution considerably stronger than that used in Raoult's freezing method. The advantage, however, of having a mercurial column sensibly constant for long intervals of time, renders it possible to make the observations with great accuracy.

To allow for the slight changes which take place in the boiling

point of water, under the conditions mentioned, I propose, hereafter, to use two pieces of apparatus, one of which shall contain water to be kept constantly in use, the observation of the boiling point of water and of the substance under examination being made at the same time. It is hardly necessary to mention that in such a case the thermometers employed would be carefully compared and the readings corrected accordingly. It is further necessary that the source of heat be sensibly constant, and we secure this by using a lamp with a certain sized flame which remains unchanged throughout the whole series of observations. For assistance in the analytical data obtained I am indebted to Mr. W. H. Krug.

September 14, 1889.

A NEW PROCESS FOR DETERMINING THE PERCENTAGE OF FAT IN MILK, CREAM, OR SKIM-MILK.

BY C. B. COCHRAN,

Inspector of Foods, Penna. State Board of Agriculture.

The essential parts of the apparatus which any chemical laboratory would not furnish are, a boiling flask and a flask provided with a side tube and a graduated terminal tube. For the sake of convenience, I have named this flask the fat indicator.

The chemicals used are clean commercial sulphuric acid, glacial acetic acid, and washed ethyl ether.

The following directions will enable anyone to carry out the analysis. The milk to be tested should be at a temperature between 50°–70° F. Fill a 5 cc. pipette with the thoroughly mixed milk to the mark on the small tube, then deliver the milk into one of the boiling flasks, blowing out the last drop from the pipette. Add from 2½ to 3 cc. of glacial acetic acid and an equal amount of sulphuric acid. (For rapid work the acids are measured in a pipette, to which a rubber bulb is attached of such a size that when the air is driven out the proper amount of acid is taken up.) Mix the milk and acids thoroughly by shaking, then suspend the flask in boiling hot water, and allow to boil gently for 10 or 15 minutes, or until the contents have turned a dark coffee color. During the boiling process take each flask from boiler

once or twice and shake. If the flask is not shaken so as to keep the milk and acids thoroughly mixed, the fat may be darkened by action of the sulphuric acid. There is very little danger of error in this stage of the work, as less than ten minutes boiling will usually suffice; or, if the time taken is longer than 15 minutes, no harm is done. Vigorous boiling, if very much prolonged, however, may cause the fat to blacken and separate in minute globules, but this would only occur under gross mismanagement. When the boiling is finished, cool the flask by standing in cold water. As soon as the contents of the flask are cooled, add 4 cc. of ether, shake, and then again suspend the flask in boiling water for 10 or 15 minutes, or until contents have ceased bubbling. A layer of clear fat is now found floating on the liquid in the flask. Now pour the contents of the boiling flask into the side tube of the fat indicator, rinse the boiling flask with hot water and pour the rinsings into the fat indicator; then deliver boiling hot water from a wash bottle, or vessel provided with a very fine spout, into the side tube of fat indicator, thus raising the fat into the graduated tube, where its volume is read and the corresponding per cent. of fat found from the table as given at the end of this description. Before using the fat indicator, it should be rinsed out with boiling hot water, otherwise the fat may adhere to the cool sides of the flask.

The fat indicator possesses two advantages over Short's tubes —1st, The fat column can be held stationary; 2nd, The graduated tube can be made of small caliber and the fat column more accurately read. The tube is graduated in fiftieths of a cc., and the marks are so far apart as to allow of the reading of $\frac{1}{100}$ of a cc. without difficulty.

If the opening where the side tube joins the bowl of the fat indicator is too large, a globule of fat may find its way into the side tube. It can, however, easily be driven back into the bowl by blowing gently down the side tube. This must be done, however, before the fat is raised in the graduated tube.

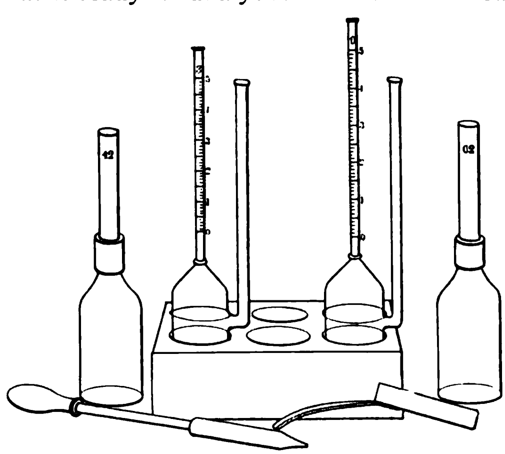
The entire operation can be performed in the fat indicator, but in this case a little more time is required. By use of the boiling flask, the ether is rapidly boiled off. The diameter of the bowl of this flask is so great that the fat does not form a complete layer

over the surface of the liquid in the flask, and hence, does not impede the rapid removal of the ether.

The author's first experiments were made in the tall form of Short's tube, but it was found to be very difficult to boil off the ether in these tubes; but when flasks of greater diameter and wider necks were substituted, this difficulty was overcome.

This method is now in use in upwards of fifty creameries, most of which are located in southeastern Pennsylvania. With the largest and most improved forms of apparatus, sixty tests can be made in from two to three hours. A copper boiler, having a perforated pipe extending the entire length, for the admission of steam to heat the water, and provided with a movable rack holding sixty boiling flasks, are used in creameries having a large number of patrons. This boiler is also provided with an overflow pipe and a spigot for drawing off hot water to raise the fat in the fat indicator. When the contents of the boiling flasks are to be cooled for the addition of the ether, the rack containing the flasks is removed from the copper boiler and stood in a copper cooling pan containing cold water.

For fast work, the milk is measured in a deep and narrow nickel dipper, instead of a pipette. This is not quite so accurate as the pipette, but it allows greater rapidity. The samples can be taken directly from the weighing can and poured from the dipper into the boiling flasks ready for analysis. The time needed to measure samples is thus saved. The illustration shows two boiling flasks, two fat indicators, a pipette with rubber bulb for measuring acids, and a nickel dipper for measuring milk.



The following table gives per cent. of fat corresponding to measures on the graduated tube of fat indicator :

Measures of Fat.	Per cent. of Fat.	Measures of Fat.	Per cent. of Fat.	Measures of Fat.	Per cent. of Fat.
1	.346	13½	4.67	22½	7.78
2	.692	14	4.84	23	7.95
3	1.038	14½	5.01	23½	8.13
4	1.384	15	5.19	24	8.30
5	1.730	15½	5.35	24½	8.47
6	2.076	16	5.53	25	8.65
7	2.422	16½	5.70	25½	8.83
8	2.768	17	5.88	26	9.00
8½	2.931	17½	6.05	26½	9.17
9	3.114	18	6.23	27	9.34
9½	3.287	18½	6.40	27½	9.51
10	3.46	19	6.57	28	9.69
10½	3.63	19½	6.74	28½	9.86
11	3.80	20	6.92	29	10.03
11½	3.97	20½	7.09	29½	10.20
12	4.15	21	7.26	30	10.38
12½	4.32	21½	7.43		
13	4.50	22	7.61		

This table has been constructed to give results corresponding to those obtained by Adams' method or Hehner & Richmond's formula. The results are higher than those obtained by the old gravimetric methods. A few experiments have shown this method to give results very nearly one-quarter of one per cent higher than Sohxlet's areometric method.

Experiments made in the Hanover State Laboratory under Dr. Skalweit showed that Adams' method gave results $\frac{23}{100}$ of 1 per cent. higher than the areometric method.

The outlines of this method were first published in March, 1889. Previous to this date, I had convinced myself of its complete accuracy by an extended series of comparitive tests. These, together with the results of other chemists, I hope to publish in the near future.

In calculating the above table, the sp. gr. of butter fat at 150° F. was used, hence the fat column, theoretically, should be read at about this temperature. In a comfortably warm room the fat will be near enough to this temperature if read as soon as possible after raising. No contraction of the fat column can be noticed in small differences of temperature.

ELECTROLYTIC SEPARATIONS.*

BY EDGAR F. SMITH AND LEE K. FRANKEL.

I. SEPARATION OF CADMIUM FROM COPPER.

The results obtained in the electrolysis of cyanide solutions of mercury and copper, as well as the knowledge gained from our experiments in separating cadmium from zinc, induced us to try whether it would be possible to effect the separation of cadmium from copper in cyanide solution. The only electrolytic method proposed for the separation of these metals is that recommended by Smith (*Am. Chem. Journal*, **2**, 42), in which solutions containing a definite amount of free nitric acid were employed. We experienced no difficulty in making a complete separation, after ascertaining the proper current strength. The conditions most favorable to the separation were essentially the same as those recorded in our former papers, viz: a dilution of 200 cc.; 5.5 grammes of potassium cyanide in each experiment, and a current generating .28 cc. oxy-hydrogen gas per minute. Time sixteen to twenty hours. The results obtained were as follows:

I.

Cadmiun present.	*Cu present.*	*Cadmium found.*
.2426 grs.	50 per cent.	.2420

II.

.2318	50 per cent.	.2331
	"	.2309
	"	.2315

*Read at the Meeting of the Chemical Section, Franklin Institute, June 18, 1889.

III.

Cadmium present.	*Cu present.*	*Cadmium found.*
.1024	100 per cent.	.1028
	"	.1019
	"	.1920
	"	.1032
	"	.1033
	"	.1034
	"	.1014

IV.

.2046	100 per cent.	.2034

Copper was not found in any of the deposited cadmium, nor did we discover any of the latter metal in the filtrate containing the copper. The cadmium deposit invariably showed the gray color characteristic of it.

II. ACTION OF THE CURRENT UPON METALLIC SULPHOCYANIDES.

It is well known that in the electrolysis of the sulphate or nitrate of manganese the oxide of this metal separates upon the positive pole. We have found that if an excess of potassium sulphocyanide be present no such deposition occurs, but, on the contrary, the metal itself will separate as a grayish white, compact deposit upon the negative pole or the vessel attached to it. The addition of a sulphocyanide to the manganese solution undergoing electrolysis, after the oxide has already separated, will cause the solution of the latter. The current necessary for the deposition of metallic manganese, under the conditions mentioned above, should be feeble. The metallic deposit is inclined to rapidly oxidize, and whether manganese can be successfully determined in a quantitative way after this manner will depend very much upon whether we can prevent its oxidation during the drying process.

Nickel, cobalt, iron and several other metals separate very rapidly from cold sulphocyanide solutions under the influence of a weak current.

CHEMICAL LABORATORY, UNIV. OF PA.,
PHILADELPHIA, June 18, 1889.

ON THE DECOMPOSITION OF ORGANIC MATTER IN WATER.

By Mark Powers.

The experiments herein detailed were undertaken with a view to obtain more exact information in regard to the decomposition of organic matter in water. While we have experimental evidence bearing on many points in connection with the subject, a review of this evidence will show that in many cases conclusions have been drawn which can be scarcely justified by the facts already known. We find many conflicting statements in the literature of the subject, and because of this fact and of the undoubted importance of the question, I have thought it worth while to repeat certain experiments, with such modifications as would be most likely to reveal the truth in each instance. I have also endeavored to obtain further facts to aid in the solution of the problem.

The organic matter dissolved by water or held in suspension, is derived from the decay of the proximate constituents of plants and animals, and may be taken up at any stage of this process. The compounds which enter into the composition of this matter are more or less unstable, undergoing change with ease, and very little is known concerning their individual properties. The element of danger does not arise from any inherent toxic properties of these compounds, but from the fact that water containing such matter serves as an excellent means for the development and distribution of disease germs. Certain products of decay are much more harmful than others in this respect, and certain conditions lend special power to bring about harmful results.

Simple oxidation, putrefaction, and the different varieties of fermentation, are the chief agencies which effect this decomposition. Carbon dioxide, water and ammonia are the principal ultimate decomposition products of organic matter. Ammonia may undergo the further transformation into nitric acid. The intermediate products are quite numerous and complex. Carbohydrates yield by fermentation a large number of alcohols, aldehydes, ethers and acids of the fatty series. Vegetable matter in contact with moist soil is converted into a brownish-black mass or humus which contains a great many unstable organic compounds. The fats yield glycerine and fatty acids. Fæcal matter contains un-

digested food and numerous decomposition products of albuminous matter and other proximate constituents of food. The albuminoids, including egg albumen, casein, blood-fibrin, peptones, etc., and the closely related bodies, such as gelatin and chondrin, yield a large and very important class of compounds. The principal products are the amido derivatives of the fatty acid series—leucine, glycocoll, aspartic and glutamic acids, etc.; members of the fatty acid series—formic, acetic, propionic, bytyric, etc.; the compound ammonias—methylamine, ethylamine, propylamine, etc.; members of the aromatic group—phenol, cresol, skatol, indol, tyrosin, hydroparacumaric acid, alphatoluic acid, etc. These compounds yield simpler products by further decomposition. Albuminous matter enters largely into the composition of the animal body, and is found in small quantity in nearly all parts of the plant, with the exception of the seed, where it occurs more abundantly. It is the decomposition of these highly complex nitrogenous compounds in water that introduces the element of danger, as the organic nitrogen results almost entirely from the decomposition of albuminous matter and derived products. The amount of free ammonia and nitric acid indicates with more or less accuracy the extent of changes which have already taken place. In the following experiments, an attempt has been made to furnish data from which more accurate conclusions may be drawn concerning the organic matter in water, by the study of individual deportment in case of the principal constituents of this matter. Each compound when subjected to the tests decomposes in a manner peculiar to itself.

TOTAL CONVERSION OF THE NITROGEN OF ALBUMEN INTO THE FORM OF AMMONIA.

Wanklyn's statements upon this subject are rather indefinite. This author mentions experiments of his own by which almost all of the nitrogen of albumen is converted into the form of ammonia, but does not give the details of his work. He was at first inclined to believe that the ammonia obtained was a definite portion of the total amount. According to other chemists, only a fraction of the total nitrogen of albumen will yield ammonia when boiled with permanganate solution. The details of my own experiments are given for the purpose of showing under what cir-

cumstances the total nitrogen of albumen is evolved in the form of ammonia.

Fresh White of Egg Solution No. 1.—Containing 30 gms. of fresh white of egg dissolved in 2500 cc. water. 10 cc. of this solution were diluted to 500 cc., 5 cc. of a strong solution of Na_2CO_3, free from ammonia, were added, and the mixture distilled. Four distillates of 50 cc. each were removed for the estimation of "free ammonia," then 50 cc. of a solution containing 8 gms. potassium permanganate and 200 gms. potassium hydrate per liter were added, and four more distillates of 50 cc. each were nesslerized for the "albuminoid ammonia." By continued boiling of the same portion of this solution with permanganate mixture on successive days, the total nitrogen in egg albumen appears in the form of ammonia. Before each distillation 200 cc. of pure water was added to the contents of the retort. The retort was kept tightly stoppered during intervals between successive distillations in order to preserve the solution free from atmospheric impurities. Care was taken to wash the condenser thoroughly with water free from ammonia, before being used. An aliquot part of the first distillate was taken for comparison with standards where depth of color would interfere with accuracy.

In calculating the amount of ammonia which by theory should be obtained from a certain weight of white of egg, I have taken the values usually given (see König's Nahrungsmittel), viz.: 12.5 per cent. of albumen with 15.7 per cent. of nitrogen. Under theory, the values so obtained are given below.

	Free Ammonia. Mgms. NH_3.	*Albuminoid Ammonia.* Mgms. NH_3.
First Distillation	.0675	1.420
Second "		.270
Third "		.146
Fourth "		.095
Fifth "		.061
Sixth "		.037

Total amount, 2.029 mgms. NH_3
Theory, 2.85 " "

Solution of Egg Albumen No. 2.—Containing 48.710 gms. fresh white of egg dissolved in 2500 cc. water. 10 cc. of this solution were taken for distillation. The same precautions as given above were taken with this series of distillations.

	Albuminoid Ammonia. Mgms. NH_3.
First Distillation	2.620
Second "	.475
Third "	.210
Fourth "	.208
Fifth "	.153
Sixth "	.122
Seventh "	.095
Eighth "	.099
Ninth "	.075
Tenth "	.070
Eleventh "	.060
Twelfth "	.050
Thirteenth "	.038

Total amount, 4.275 mgms. NH_3
Theory, 4.63 " "

Egg Solution No. 3, made by dissolving dried albumen in distilled water, was decomposed much more slowly.

It will therefore be observed that it is possible by continued distillations with alkali-permanganate solution to convert the total nitrogen of albumen into the form of ammonia. Egg solution No. 2 yields nearly 95 per cent. of this element. The decomposition is, however, very slow under the conditions of the experiment. The rate depends upon a variety of circumstances,—the concentration and quantity of permanganate solution, shape and size of retort, rapidity of distillation, etc. In egg solution No. 1 about 50 per cent. of the nitrogen in the form of ammonia was evolved during the first distillation; in solution No. 2, a larger proportion, while from a solution of dry albumen in water, only a little over 35 per cent. of the total amount was obtained.

Decomposing Solution of Egg Albumen.

Fresh white of egg solution No. 1 was allowed to putrefy, and the process of decomposition was traced from the beginning by means of the amount of free and albuminoid ammonia evolved. 10 cc. were taken for each test. Precautions were taken to keep the room as free as possible from ammonia vapor or other substances liable to contaminate the solution. I am not aware that anyone has traced the process of decomposition of albuminoid material in this way. The conditions simulate those that exist in natural waters. Tiemann and Preusse have traced the process

of decomposition of egg albumen by means of the oxidation test with standard solution of potassium permanganate, but only for a short time.

	Free Am. mgms. NH_3	Alb. Am. mgms. NH_3		Free Am. mgms. NH_3	Alb. Am. mgms. NH_3
Feb. 19	.072	1.420	May 21	1.680	.541
" 26	.067	1.120	" 28	1.785	.496
Mar. 5	.110	1.113	June 4	1.904	.455
" 12	.442	1.068	" 11	1.965	.444
" 19	.623	1.055	" 18	2.035	.413
" 26	.890	1.015	" 25	2.206	.352
April 2	.940	.958	July 2	2.127	.355
" 9	.978	.838	" 9	2.129	.304
" 16	1.013	.763	" 16	2.140	.298
" 23	1.327	.700	" 23	1.990	.320
" 30	1.375	.680	" 31	2.092	.320
May 7	1.525	.595	Aug. 13	1.915	.322
" 14	1.635	.578	" 27	2.124	.332

Putrefying solution of egg albumen yields a gradually increasing quantity of free ammonia, while the albuminoid ammonia regularly diminishes. The free ammonia plus the albuminoid ammonia is an increasing quantity, and near the latter part of the time occupied by experiment is above 90 per cent. of the total nitrogen.

UREA.

There are perhaps more conflicting statements in regard to the decomposition of this compound than any other. Wanklyn states that urea, when quite pure, may be boiled for a long time with alkalies without evolving a trace of ammonia, and again in presence of permanganate and excess of potash, urea is doubtless decomposed, but it yields no ammonia. Tiemann obtains, on the other hand, the total nitrogen in the form of ammonia. Other chemists assert the possibility of obtaining the total amount. In one of Wanklyn's experiments, by boiling for a long time with

strong permanganate mixture, only 22 per cent. could be obtained. The details of my own experiments with this compound are given below.

The total nitrogen of urea is converted into ammonia, if a simple solution in water is boiled during a sufficient length of time. The decomposition is more rapid if strong solution of alkali-permanganate be added.

Experiment 1.—2.5 mgms. urea were dissolved in 500 cc. water, and 5 cc. of a strong solution of Na_2CO_3 added, and the mixture distilled for free ammonia, after which 50 cc. of permanganate solution were added for the albuminoid ammonia.

Free ammonia $= .028 + .014 + .012 + .011$ mgms. $NH_3 = 4.5\%$ NH_3

Albuminoid ammonia $= .033 + .022 + .021 + .027 + .056$ mgms. $NH_3 = 11\%$ NH_3

Total $= 15.5\%$ of theoretical yield of NH_3

Experiment 2.—½ mgm. urea,—otherwise same as in experiment 1.

Free ammonia $= .022 + .012 + .0015 + .001$ mgms. $NH_3 = 12\%$ NH_3

Albuminoid ammonia $= .022 + .007 + .005 + .003$ mgms. $NH_3 = 13\%$ NH_3

Total $= 25\%$ of theoretical yield of NH_3

Experiment 3.—½ mgm. urea,—100 cc. permanganate solution.

Free ammonia $= .022 + .012 + .0015 + .0015$ mgms. $NH_3 = 13\%$ NH_3

Albuminoid ammonia $= .020 + .012 + .012 + .0125$ mgms. $NH_3 = 19\%$ NH_3

Total $= 32\%$ of theoretical yield of NH_3

Experiment 4.—1 mgm. urea,—2 gms. potassium permanganate and 40 gms. potassium hydrate added for albuminoid ammonia.

Free ammonia $= .034 + .025 + .012 + .0075 + .005$ mgms. $NH_3 = 16\%$ NH_3

Albuminoid ammonia $= .068 + .023 + .021 + .0225 + .030$ mgms. $= 28\%$ NH_3

Total $= 44\%$ of theoretical yield of NH_3

Experiment 5.—1 mg. urea in 450 cc. water with 50 cc. permanganate solution added. Boiled during ten hours in retort with inverted condenser and bulbs containing dilute solution of sulphuric acid attached at other end. Two sets of bulbs were employed and blank experiments were made in order to test the connections of the apparatus, and to ascertain if any other sources of error might be present in the experiment. The amount of ammonia obtained in the blank trial was so small as to be neglected altogether. The albuminoid ammonia obtained from urea was 78 per cent. of the total amount. By distilling off four distillates of 50 cc. each for four successive times, the remaining 22 per cent. of the ammonia was obtained.

Experiment 6.—2 mgms. urea. Boiled solution during 20 hours,—otherwise same as in experiment 5. Albuminoid ammonia obtained, 101 per cent.

Experiment 7.—2 mgms. urea. Boiled during 22 hrs. in 500 cc. water only. Ammonia obtained, 95 per cent. of the theoretical amount.

In this connection, the results obtained by Dr. Charles Smart are of interest (Sanitarian, Nov., 1886). He states, "Since 1 mgrm. urea in 500 cc. of water gives a persisting and equable evolution of .01 mgm. of ammonia when distilled alone or with sodium carbonate and an evolution of .02 in each measure when subsequently treated with alkaline permanganate, a water sample which yields such results must have contained urine equivalent to, at least, 1 mgrm. of urea," and again, "a water which yielded in successive distillates of 50 cc. each .47, .25, .15, and .15 mgms. of free ammonia, and afterward .54, .34, .32 and .32 mgms. of albuminoid ammonia might be set down as having contained urine equivalent to, at least, 15 mgms. of urea in the 500 cc. of water used in the experiment." If these statements expressed the real facts in the case, the observation would doubtless be of great value. The equable evolution refers to the third, fourth and subsequent distillates, and the results are in direct proportion to the amount of urea present. The experiments I have made with urea prove that no set or combination of conditions can concur to produce such an evolution of ammonia. The decomposition of urea takes place very slowly, even with strong solution of permanganate, but wide variations result from apparently slight

changes in the conditions of the experiment. My experiments were made in as nearly uniform a manner as possible, with reference to the time in collecting distillates. Experiments 2 and 3, with one-fifth as much urea as in experiment 1, gave, on the other hand, nearly twice the quantity of total ammonia, and furthermore, a comparison of the third, fourth, and subsequent distillates, in each case reveals the fact that the amount of albuminoid ammonia evolved, is from two to six times the quantity of free ammonia, and is not in the constant ratio of 2 : 1. The evolution of ammonia from urea is quite variable, and being associated in natural waters with other nitrogenous compounds of unknown character, there is no trustworthy evidence that will determine the quantity of the substance with the degree of precision stated by this author.

OTHER NITROGENOUS SUBSTANCES.

In the following table I have given the results of my experiments, showing how much of the total nitrogen can be evolved as ammonia by the ordinary Wanklyn method, from an important class of nitrogenous compounds.

1 mg. Substance.	Mgms. NH_3 Evolved.	Theoretical Amount.	1 mg. Substance.	Mgms. NH_3 Evolved.	Theoretical Amount.
Leucine $C_6H_{13}NO_2$	.130	.130	Asparagine $C_4H_8N_2O_3+H_2O$	.200	.226
Glycocoll $C_2H_5NO_2$	.225	.226	Quinine Sulphate $(C_{20}H_{24}N_2O_2)_2H_2SO_4+8Aq$	.040	.080
Aspartic Acid $C_4H_7NO_4$	.125	.130	Quinidine Sulphate $(C_{20}H_{24}N_2O_2)_2H_2SO_4+2Aq$	.043	.071
Indol C_8H_7N	.144	.145	Strychnine $C_{21}H_{22}N_2O_2$	.097	.101
Skatole C_9H_9N	.120	.130	Trimethylamine Chlor. $C_3H_7NH_2HCl$	.180	.178
Tyrosine $C_9H_{11}NO_3$	.094	.094	Aniline Chloride $C_6H_5NH_2HCl$	.060	.131
Urea CH_4N_2O	.060	.058	Albumen	.149	.157
Hippuric Acid $C_9H_9NO_3$	.090	.095			

In this list of compounds, the intention has been to include the important decomposition products of albumen, such as leucine, tyrosine, aspartic acid, glycocoll, indol, and skatole, as the greater portion of the organic nitrogen of natural waters occurs as a constituent of derived products from albuminous matter. Tiemann and Preusse have obtained the total amount of nitrogen in leucine, tyrosine and aspartic acid in the form of ammonia. My own experiments gave similar results in case of each substance mentioned, the total nitrogen being obtained by the first distillation.

Hippuric acid decomposes in a very peculiar manner. 3 mgms. of this acid were dissolved in 500 cc. of water, 200 cc. were distilled for free ammonia, 50 cc. permanganate solution added, and the albuminoid ammonia obtained was .02, .03, .042, .05, .043 mgms. in the different distillates; a second distillation gave .065, .008, .002 mgms., thus completing the evolution of the total nitrogen in the form of ammonia.

Wanklyn states that quinine, quinidine, strychnine, etc., yield one-half their nitrogen in the form of ammonia. I have obtained similar results in case of quinine and quinidine, but the sample of strychnine tested gave nearly the whole amount.

THE OXIDATION OF ORGANIC MATTER IN WATER BY MEANS OF PERMANGANATE SOLUTION.

The extent to which a large number of important organic compounds are oxidized, especially the principal decomposition products of albumen, is shown in the following table. The methods of Schulze and Kubel are compared by giving the results of each. In Schulze's method 100 cc. of water containing a definite amount of the compound is boiled during 10 minutes with constant excess of standard potassium permanganate solution (containing 320 mgms. per liter) and a definite quantity of pure sodium hydrate, while in Kubel's method the substance is boiled during 10 minutes in solution acidified with sulphuric acid. By either method the amount of oxygen consumed varies from a mere trace to almost the total amount necessary for complete oxidation. The correction necessary in Schulze's method was determined by making blank experiments.

1 mg. Substance.	Schulze's Method. Mgms. Oxygen Consumed.	Kubel's Method. Mgms. Oxygen Consumed.	Mgms. Oxygen required for Complete Oxidation.	1 mg. substance.	Schulze's Method. Mgms. Oxygen Consumed.	Kubel's Method. Mgms. Oxygen Consumed.	Mgms. Oxygen required for Complete Oxidation.
Leucine . . . $C_6H_{13}NO_2$	.216	.208	2.015	QuinineSulphate $(C_{20}H_{24}N_2O_2)_2H_2SO_4$ +8aq	.480	.820	1.798
Tyrosine . . . $C_9H_{11}NO_3$	1.224	1.320	1.812	Quinidine Sulph. $(C_{20}H_{24}N_2O_2)_2H_2SO_4$ +2aq	.416	.752	2.046
Glycocoll . . . $C_2H_5NO_2$	.100	.100	.960	Cinchonine Sul. $(C_{19}H_{22}N_2O)_2H_2SO_4$ +2aq	.440	.744	2.127
Aspartic Acid . $C_4H_7NO_4$	.368	.368	.902	CinchonidineSul $(C_{19}H_{22}N_2O)_2H_2SO_4$ +6aq	.400	.640	1.964
Aspargine . . . $C_4H_8N_2O_3+H_2O$	.112	.08	.960	Strychnine Sul. $(C_{21}H_{22}N_2O_2)_2H_2SO_4$ +6aq	.872	1.048	1.867
Indol . . . . . C_8H_7N	1.952	2.080	2.666	Morphine Chlor. $C_{17}H_{19}NO_3HCl+3aq$	1.016	1.062	1.726
Skatole . . . . C_9H_9N	1.696	1.720	2.748	Tannic Acid . . $C_{14}H_{10}O_9+2aq$	.768	.797	1.073
Benzoic Acid . $C_7H_6O_2$	.026	.096	1.967	Citric Acid . . . $C_6H_8O_7$	.504	.490	.750
Urea . . . . . CH_4N_2O	trace	trace	.800	TartaricAcid . . $C_4H_6O_6$	.368	.432	.533
Hippuric Acid $C_9H_9NO_3$	.224	.228	1.743	Starch . . . . . $C_6H_{10}O_5$	.540	.544	1.185
Trimethylamine Chloride $C_3H_7NH_2HCl$	.024	.032	1.761	Cane Sugar . . $C_{12}H_{22}O_{11}$	.628	.636	1.123
AnilineChlor. . $C_6H_5NH_2HCl$	1.312	1.320	1.916	Lactose . . . . $C_{12}H_{22}O_{11}+H_2O$	.640	.640	1.066
Salycilic Acid . $C_7H_6O_3$	1.392	1.480	1.623	Glucose . . . . $C_6H_{12}O_6$	.650	.648	1.066

From the results of these experiments, Kubel's method, being simpler, has the preference. The results obtained by the two methods are in most cases approximately the same. A large number of the above compounds require over one-half of the amount of oxygen necessary for complete oxidation. In several instances these results accord with those given by Tiemann and Preusse, while in others, different figures were obtained. Without doubt, the application of the principle of oxidation, according to the methods detailed above, yields more satisfactory informa-

tion than any other so far proposed. Dr. Frankland states that cane sugar and starch do not require so much as one hundreth of the oxygen necessary for total oxidation; by Kubel's method, the quantity is over one-half the amount required. In other cases the oxidation would be slight at the ordinary temperature, while at the boiling point of water a large amount of oxygen would be consumed.

In conclusion, several statements may be made in reference to the results obtained by these experiments.

Albumen, urea, and some other compounds, will evolve their total nitrogen in the form of ammonia by prolonged boiling with strong solution of alkali-permanganate.

No definite portion of the total nitrogen of these compounds is obtained in the first distillation, the amount depending upon a number of conditions, of which the quantity of substance used and the rapidity of distillation are the chief factors that vary the result. Albumen gave in one experiment by the first distillation 1.42 mgms. ammonia, while the total amount was 2.8 mgms.; in a second experiment 2.63 mgms.,—total, 4.63 mgms. In one instance thirteen successive distillations were made, collecting four distillates of 50 cc. each, the total amount obtained being about 95 per cent. Egg albumen undergoing putrefaction yields a gradually increasing quantity of free ammonia, while the albuminoid ammonia decreases in a regular manner.

The principal decomposition products of albumen give up readily their total nitrogen in the form of ammonia. Indol and skatole are carried over in the distillation of free ammonia, and give a milkiness to the solution when nesslerized. This fact may account in part for the "haziness" frequently observed in nesslerizing.

2.5 mgms. urea gave of free and albuminoid ammonia 15.5 per cent., while ½ mg. yielded 25 per cent. Urea was boiled 10 hours with strong solution of alkali-permanganate, but not completely decomposed. Complete decomposition was effected, however, by boiling 2 mgms. during 20 hours with 50 cc. of permanganate solution. On the other hand, 2 mgms. of this compound were almost completely decomposed by boiling in water alone for same length of time. The amount of ammonia evolved from urea

by the ordinary method depends upon the amount of this substance in solution, a smaller quantity yielding a relatively larger quantity of both free and albuminoid ammonia.

Hippuric acid yields more albuminoid ammonia in the last distillate than in the first. It is, however, completely decomposed by a second distillation.

Quinine and quinidine yield apparently about one-half of their total nitrogen in the form of ammonia. The sample of strychnine tested gave nearly the total amount.

The amount of oxygen consumed varies from a mere trace to almost the total amount required by theory. About one-half the number of compounds studied required from one-half to seven-eighths of the total amount of oxygen. Indol, skatole and tyrosine strongly reduce the permanganate solution. A large number of organic compounds containing no nitrogen, such as the sugars, alcohols, vegetable acids, etc., are stronger reducing agents than some nitrogenous compounds which may be considered more dangerous.

No compounds were examined except urea, which did not consume more or less oxygen.

For many valuable suggestions while engaged in the study of these compounds, I am deeply indebted to Dr. John H. Long, in whose laboratory these experiments have been made.

CHICAGO, Oct. 1, 1889.

A METHOD, IN PART, FOR THE SANITARY EXAMINATION OF WATER, AND FOR THE STATEMEMT OF RESULTS, OFFERED FOR GENERAL ADOPTION.

As has been mentioned in previous numbers of this Journal, the Chemical Section of the American Association for the Advancement of Science has taken into consideration the matter of attempting to establish some uniformity in the methods of analysis of potable waters, and of stating the results thereof, and a committee was appointed to consider this subject and report upon it. At the meeting of the Section this year, in August, this committee presented a report, recommending for provisional adoption the following method for the sanitary examination of water so far

as refers to nitrogen compounds, and oxygen-consuming capacity, and for stating all the results of the usual sanitary analysis; the report was accepted, together with the further recommendations of the committee that directions for the method be printed and circulated among chemists, with the request for opinions as to its suitability for general adoption, and the results of experience in its use.

The method of examination thus presented embodies as far as practicable the results of the investigations of Professor Mallet, Dr. Smart and Dr. Tanner, given in the report of the United States Board of Health for 1882, and is in accord, also as far as practicable, with the best practice of water analysts in this country in so far as it has been possible to ascertain by correspondence what that practice is.

I. "FREE" AND "ALBUMINOID" AMMONIA.

Presuming that every one interested in the sanitary examination of water possesses or has access to Wanklyn's "Water Analysis," the directions for these two determinations will be given only to such an extent as may be necessary to indicate clearly the modifications of his method that are recommended.

a. The Reagents.—The Nessler Reagent.—"Dissolve 35 gms. of potassium iodide and 16 gms. of mercuric chloride each in a small quantity of water, add the mercuric solution to that of the iodide till a faint show of excess is manifested, then add to the mixture a solution of 160 gms. of potassium hydrate in 800 cc. of water, and make up to one liter; finally, sensitize the solution by the addition of a few drops of a cold saturated solution of mercuric chloride, and put it away, to become clear, in the stock bottle in which it is to be kept.*

The Standard Solution of Ammonium Chloride.—Use ammonia free water for this solution, and the pure and dry salt.

The Alkaline Permanganate Solution.—Dissolve the 8 gms. of permanganate and 200 gms. of potassium hydrate in 1250 cc. of water, and boil down to 1000 cc.

The Solution of Sodium Carbonate.—This is to be itself tested for ammonia as below, even if made from recently heated salt.

*Mallet: Report U. S. Board of Health, 1882, p. 279.

The Ammonia Free Water.—This is to be tested for ammonia. In preparing it by redistillation, the operation is much facilitated by the addition of a little phosphoric acid to the water in the retort.

b. The Apparatus.—The neck of the retort must pass within the mouth of the condenser tube to the distance of 3 or 4 cm., drawing it down smaller for this purpose if necessary, and a tight rubber connection between the two is to be made; the retort should have a good fitting glass stopper.

c. The Operation.—Two hundred (40) cc.* of distilled water together with 10 (2) cc. of the solution of sodium carbonate are distilled down to about 100 (20) cc. in the retort in which the analysis is to be conducted, and the last portion of 50 (10) cc. nesslerized, to assure freedom from ammonia. Then 500 (100) cc. of the water to be examined are added and the distillation is carried on at such a rate that about 50 (10) cc. are collected in each succeeding ten (2) minutes, and till a 50 (10) cc. measure of distillate is obtained containing only an inappreciable quantity of ammonia. In nesslerizing five minutes are to be allowed for the full development of the color; after this no change takes place for many hours.

Now throw out the contents of the retort, rinse it thoroughly, put in 200 (40) cc. of distilled water and 50 (10) cc. of the permanganate solution, distil down to about 100 (20) cc. and nesslerize the last portion of 50 (10) cc. to make sure of freedom from ammonia; add another portion of 500 (10) cc. of the water under examination, and proceed with the distillation and nesslerizing as with the first portion.

The difference between the "free" ammonia of the first operation and the total ammonia of the second is to be taken as the "albuminoid" ammonia.

The details of the evolution of ammonia in the successive distillates from each portion of water should be noted, even if not

*In view of the apparently very general practice in this country of taking 500 cc. of water for the analysis the committee did not deem it wise to recommend, now, the taking of 100 cc. given by Wanklyn as an alternate method, notwithstanding that to several of its members this latter method seemed more desirable; but to suggest trials of it in comparison with the more cumbrous 500 cc. method, the figures are given in parentheses for the quantities to be operated with, 100 cc. of the water being taken.

reported. (A large proportion of ammonia in the first distillate, much less in the second, and a tolerably uniform proportion in the succeeding ones, from both portions of the water, with a ratio of one of "free" to two of "albuminoid" ammonia, indicates urea, or recent contamination with urine (Dr. Smart).

Loss of ammonia from imperfect condensation being easily possible, this danger should be reduced to a minimum by the use of as large a condensing surface as practicable, and a rapid flow of cold water through the condenser.

All this ammonia work is to be done in a room free from contamination of the air with ammonia, and with reagents whose purity is carefully assured.

2. OXYGEN-CONSUMING CAPACITY.

"Prepare a solution of potassium permanganate containing 0.1 mgm. of available oxygen to 1 cc. and a solution of oxalic acid of such strength as to decompose the permanganate solution, volume for volume, the strength being redetermined from time to time. The water used should be purified by distillation from alkaline permanganate.

To 200 cc. of the water to be examined, in a 400 cc. flask, add 10 cc. of dilute sulphuric acid (1:3) and such measured quantity of the permanganate as will give a persistent red color; boil ten minutes, adding if necessary more permanganate in measured quantities, so as to maintain the red color; remove the flask from the lamp, add 10 cc. of oxalic acid solution to destroy the color, or more if required by the excess of permanganate, and then add permanganate, dropwise, till a faint pink tinge again appears. From the total quantity of permanganate used deduct the equivalent of the oxalic acid added, and from the remainder calculate the milligrams of oxygen consumed by the oxidizable organic matter in the water. Of course correction must also be made for nitrites, ferrous salts and hydrogen sulphide, if any of these substances are present in the water." (Dr. Smart.)

3. TOTAL NITROGEN AS NITRATES AND NITRITES.

This is determined by reduction to ammonia by the copper-zinc couple, and nesslerizing.

Take two glass-stoppered, wide mouth bottles holding 250 cc., and a piece of sheet zinc as long and about as wide as the bottles are deep from the shoulder down; clean the zinc by dipping in dilute acid and washing with water, make it into a loose coil by rolling it round a piece of large glass tube, immerse it in a 1.4 to 1.8 per cent. solution of cupric sulphate in ammonia-free water, and leave it there till its surface is well covered with a continuous layer of the black copper; lift it out carefully, cover it in a beaker with successive portions of ammonia-free water, lifting it out and draining each time, and at once put it into one of the bottles of acidified water prepared as follows:

Make 500 cc. of the water to be examined distinctly acid with oxalic acid, added in fine powder and with constant stirring so that it shall dissolve readily, and pour half of the liquid into one of these 250 cc. bottles and half into the other, and leave them stoppered, in a warm place for twenty-four hours. Then nesslerize both samples, decanting off the portions as wanted, from the precipitated earthy oxalates at the bottom of the bottles, and using double the usual quantity of Nessler solution, since the free oxalic acid has to be neutralized first by the alkali of the reagent. The proportion of ammonia may often be so large in the water in which the reduction was made by the copper-zinc couple, that only 5 or 10 cc. can be taken for each test, and made up to 50 cc. by the addition of ammonia-free water.

The difference between the results with the two portions of water gives the amount of nitrogen due to the oxidized nitrogen compounds in the water examined.

4. NITROGEN AS NITRITES.

This is determined by the Griess method. Follow the directions given by Leffmann and Beam's "Examination of Water." (P. Blakiston Sons & Co., Philadelphia.)

5. THE STATEMENT OF RESULTS.

All the results are to be stated in parts per million (milligrams per liter), and in the manner shown below:

Total solids . . . .
Chlorine . . . .
Nitrogen : expelled as ammonia on boiling with Na_2CO_3 equals "free ammonia" . . . .
expelled as ammonia only on boiling with alkaline permanganate . . . .
equals "albuminoid ammonia" . . . .
as nitrite . . . .
as nitrate . . . .
Organic matter (in terms of mgms. of oxygen consumed by one million of water) . . . . . .
Hardness . . . .
Etc . . . .

The undersigned will be glad to receive suggestions from any chemists interested in this subject, based on their own experience, that may lead to such modifications of this method as will tend to make it more widely acceptable. The substance of these communications will be duly laid before the Chemical Section of the Association at its next meeting.

It is the hope of the Committee that many trials will be made of Drown and Martin's application of the Kjeldahl nitrogen method in the sanitary examination of water; the results of experience therewith would be useful to us in any further consideration of this matter.

G. C. CALDWELL,
Chairman of Committee.

ITHACA, N. Y.

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[*The size is 8vo. when not otherwise given.*]

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REPORT OF THE COMMITTEE ON INDEXING CHEMICAL LITERATURE.*

The committee on Indexing Chemical Literature respectfully presents to the Chemical Section its seventh annual report.

During the year just closed three bibliographies have been published by the Smithsonian Institution.

A Table of Specific Gravity for Solids and Liquids. The Constants of Nature, Part I (new edition, revised and enlarged). By Frank Wigglesworth Clarke. Washington, D. C., 1888. Smithsonian Miscellaneous Collections No. 659. 8vo, pp. xi, 409.

This volume contains the specifiic gravities of 5,227 distinct substances and 14,465 separate determinations, being more than twice as many as in the first edition with supplement.

Index to the Literature of Columbium, 1801–1887. By Frank W. Traphagen. Smithsonian Miscellaneous Collections No. 663. Washington, 1888. 8vo, pp. [iv], 27.

This Index contains the three fold arrangement chronological, alphabetical by authors, and subject-matter. The abbreviations of Journal-titles are those adopted by the Committee on Indexing Chemical Literature upon whose recommendation this Index was printed.

A Bibliography of Chemistry for the year 1887 by H. Carrington Bolton. Washington, 1888. Smithsonian Miscellaneous Collections No. 665. 8vo, p. 13.

Prof. Wm. L. Dudley, Vice President of the Chemical Section of the A. A. A. S., has appended to his address on the Nature of Amalgams an Index to the Literature of the subject which will be printed in the forthcoming volume of the proceedings of the Toronto meeting.

We record the publication of the following index. Lists of patents relating to Soap and Candles in "Manufacture of Soap and Candles" by Wm. Brannt, Philadelphia, 1889.

Also:—An Index of Researches upon the production of Ammonia from Atmospheric Nitrogen. By Ezra J. Ware. Published in Proceedings Michigan State Pharmaceutical Association, 1888, H. J. Brown, Ann Arbor, Mich., Secretacy.

*From Advance Sheets of the Proceedings of the American Association for the Advancement of Science, Vol. xxxviii.

Reports of progress from several volunteer indexers have been received. Dr. Alfred Tuckerman has completed his Index to the Literature of Thermodynamics except a subject index to which he is putting finishing touches. He has also begun an Index to the Literature of the Chemical Action of Light, a subject suggested by the Chairman of your Committee.

The publication of this annual report is always followed by numerous applications for information, especially with respect to the method of indexing adopted by the Committee. These inquiries we may in some degree anticipate by repeating the statement that this Committee does not prescribe any standard, nor dictate any system for volunteers to follow. Those interested in the work of the Committee would do well to examine the six reports already published, in which will be found suggestions for systematic indexing (1885), a complete list of indexes already printed under the auspices of the Committee (1887), and a list of "Abbreviations of Titles of Chemical Journals," intended to secure uniformity in references (1887).

The choice of subjects for indexing has generally been made by the volunteers themselves; in a few instances only the topics have been suggested by the committee, at the request of co-workers. These subjects, as shown in our reports, cover a wide range of physical, inorganic and organic chemistry; the desirability of securing indexes to individual elementary substances should not be overlooked by those who contemplate offering aid. A few copies of the Report of 1887 (which includes lists as above) remain, and can be had on application to the chairman, to whom also letters of inquiry may be addressed, care of Smithsonian Institution, Washington.

H. Carrington Bolton,
Chairman.
F. W. Clarke.
A. R. Leeds (in Europe).
A. A. Julien.
John W. Langley.
Albert B. Prescott.
Chas. E. Wead.

THE SOLUBILITY OF PHOSPHATIC SLAG IN AMMONIUM CITRATE SOLUTION.

BY H. W. WILEY.

In 1882, while examining some very finely ground bone in the laboratory of Purdue University at Lafayette, Ind., I noticed a marked solubility of this fine bone in citrate of ammonia. With the ordinary coarsely ground bone no such solvent action was noticed. These facts were published in the proceedings of the Agricultural College of Indiana for the year 1882.

Recently, July 16, 1889, I received from Wm. S. McFarland, chemist at the Pottstown Steel Works, a sample of phosphatic slag called by him "cinder." This sample was from a mass of several thousand tons of this slag which the Company had prepared for market. In preparing it, they used the Cyclone Pulverizer, and by means of this instrument they had reduced the slag to an impalpable powder. The superior merits of basic phosphate, in the form of slag, over the natural phosphate rock in its action upon growing plants led me to believe that this slag might be largely soluble in the citrate solution. The slag subjected to analysis gave the following results:

Total phosphoric acid	20.20%
Citrate soluble phosphoric acid	12.25%

From the above results it is seen that in the phosphate slag we have a form of phosphoric acid equivalent in value to a first-class superphosphate. When it is remembered that this efficiency is secured without the use of sulphuric acid, and that the material is in the form of a dry powder and easily transportable, it is seen that we have in phosphatic slag, properly prepared, a valuable substitute for the ordinary superphosphates of commerce.

The analytical work was done under my direction by Messrs. Krug and McElroy.

ABSTRACTS.

APPARATUS AND REAGENTS.

Rapid Filtration.—Reginald A. Fessenden, Chem. News **60**, 102. The filter paper is folded three times (Fig 1). Folds Nos 1 and 2 are toward the reader and No 3 from him. The filter is then gathered (Fig 2) and a piece of glass rod bent at a very acute angle (Fig 3) is inserted in the cleft of the filter (Fig 4), which thus nearly ... [illegible]

Fig. 1

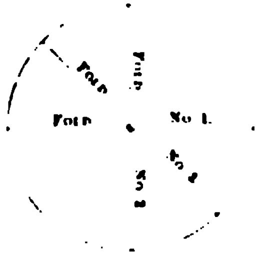


Fig. 2

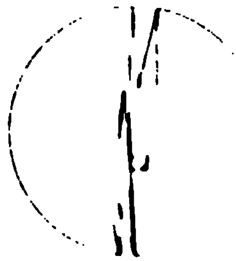


Fig. 3

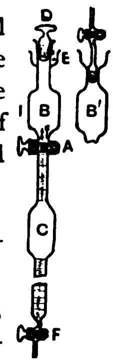
Fig. 4

Fig. 5

from the funnel by means of the glass rod, and the precipitate is easily washed off into a beaker (Fig. 5). A plate of glass (Fig. 6) ribbed on both sides, as a substitute for the glass rod, is an improvement which renders the filtration very rapid indeed.

A. H. W.

A New Gas Burette.—Greiner & Friedrichs (Chem. Zeit. **13,** 561) have designed a modification of Franke's burette, which is shown in the following cut.



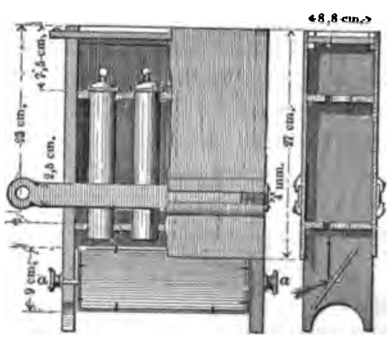
Instead of one opening, the cock A has two small cone-shaped openings, which allow the passage of the absorption solution and gas much more readily than the old burette. The lower end is supplied with one of their new stop-cocks. The stopper D may be replaced by the form shown in B if desired. S. C.

New Form of Burette.—O. Reitmair, Ztschr. angew. Chem. **89,** 361.

For titrating with permanganate solution, the author uses a burette provided with a stop-cock of the form shown in the figure. The author claims that this stop-cock needs no lubricating and is easily cleaned. When the glass faces are ground perfectly even, they move easily against each other and allow of the smallest possible drop being delivered from the burette.

J. E. W.

Colorimeter.—A. Jolles, Ztschr. angew. Chem., **89,** 365.



The apparatus consists of four glass cylinders 22 cm. high and 3 cm. in diameter, etched with 50 cc. and 100 cc. marks, and with hollow stoppers and flanges.

These cylinders are placed in a wooden case as shown in the Figure. Two slits 4^{mm} wide, opposite each other, in

the sides of the can, allow of the observation. A mirror placed in an axle under the can reflects the light up through the bottom of the cylinders. If the adjustment of the mirror is exact, the light will be the same in all four cylinders.

It is claimed that with this apparatus colors too faint to be detected with ordinary colorimeters are here plainly visible, and that the side slits answer perfectly in observing colors too dense for the light to penetrate from below. J. E. W.

INORGANIC ANALYSIS.

Estimation of Lime in Presence of Phosphoric Acid, Iron, Aluminum and Manganese.—O. Reitmair, Ztschr. angew. Chem. **89**, 357.

The author gives a review of the tests made on lime precipitated by ammonium oxalate from solutions containing phosphoric acid, iron, aluminum and manganese, and concludes that the precipitation is best when Classen's method is followed; that the precipitated calcium oxalate is free from phosphoric acid, but contains, with a large amount of manganese the oxalates of the other metals; that when this precipitate, after ignition, is dissolved in hydrochloric acid, ammonium hydrate and sulphide added, the manganese sulphide filtered off, the filtrate made acid with acetic acid, and the lime again precipitated as oxalate, the precipitate is clean and free from other oxalates. J. E. W.

The Electrolytic Method applied to Mercury, Separation from Copper.—By Edgar F. Smith and Lee K. Frankel, Amer. Chem. Journ., **11**, 4, 264. On working a solution containing .1945 gms. mercury and a large excess of KCN, a total dilution of 175 cc. and a current yielding 2 cc. gas per minute results were obtained agreeing very closely with the amount taken. The mercury was in the form of chloride. The deposited mercury was washed with water. When alcohol was used small particles of mercury were detached and lost.

A solution of copper with an excess of cyanide is not precipitated until the cyanide is decomposed. This suggests a method for separation of copper and mercury.

In several experiments on a solution containing varying quantities of copper and mercury, and using currents of different strengths, results were obtained for mercury which in most cases were less than the amount present.

Experiments on the separation of silver and copper in KCN solution gave results for silver invariably high, due to the copper precipitating with silver. The Julien storage battery was used, giving a steady current, and is highly recommended by the authors for electrolytic work. C. W. M.

On the Estimation of Sulphur in Pyrite.—G. Lunge, Ztschr. angew. Chem. 1889, 473.

The author finds three points of interest in the analysis of pyrite.

First, that in precipitating sulphur as barium sulphate from solutions of ferric chloride, the precipitate always contains iron, which, according to Jannasch and Richards, is in the form of a double sulphate of iron and barium, and that when this precipitate is washed with dilute acid to free it of iron, the wash water often contains as much as .5 per cent. of sulphur as $BaSO_4$ in solution.

Secondly, that correct results may be obtained by precipitating the iron by ammonia, in not too great excess, and washing the precipitate on the filter with hot water, and that when this precipitate of iron hydroxide is dried and fused with soda, not a trace of sulphur can be found in it.

Thirdly, that by the second method more sulphur will always be found than by the first. J. E. W.

Determination of Copper and Arsenic in Pyrites.—R. Nahnsen, Chem. News **60**, 133. (Chemiker Zeitung.) 12.5 gms. of the finely powdered sample are taken for the determination of copper, placed in a beaker of thin glass (17 centimeters high) and covered with 10 cc. water and 1 cc. conc. sulphuric acid. A porcelain capsule is placed on the beaker and nitric acid (sp. gr. 1.4) added until effervescence ceases. The liquid is then boiled over a moderately strong flame until, after frequent skaking, yellow saline particles separate out, when the paste is quickly brought into solution by warm water. The cold solution is transferred to

a 250 cc. flask, diluted to the mark, filtered through a dry filter and 200 cc. (=10 gms. of the sample) of the solution, thus freed from silica and lead are treated for several hours with a current of sulphuretted hydrogen until the liquid appears transparent, when it is filtered and washed by decantation with water, while the precipitated clots are crushed with a glass rod. The portion of the precipitate on the filter is washed back to the main mass with the least possible amount of hot water. A strong solution of sodium sulphide is now added in such quantity that the liquid, after being boiled for a few minutes shows no signs of undissolved sulphur. It is then diluted with hot water, allowed to stand in a warm place until clear, and the solution which contains the arsenic and antimony is separated from the precipitated copper sulphide by filtration. Traces of iron sulphide which may adhere to the copper sulphide are removed by hot water to which a few drops of hydrochloric acid have been added, the residue is washed until free from chlorine and the copper is determined as sulphide. The presence of bismuth or cadmium in the copper sulphide is detected by dissolving it in nitric acid and heating the solution for some time with ammonium carbonate. The precipitate formed is weighed as oxide and deducted. For determining the arsenic the dissolved sulphuretted hydrogen is expelled from the 200 cc. solution by passing a current of carbonic acid through the solution and the arsenic sulphide is dissolved out from the washed precipitate by means of ammonia. Either the volumetric method of McCay (Chem. News **48**, 7) or the gravimetric method of Fischer (Chemiker Zeitung **9**, 1613) may be used for further treatment. A. H. W.

Clarke's Soap Test: Preparation of the Standard Solution.—F. E. Nelson, Chem. News **60**, 132. 1 gm. of pure palmitic acid is dissolved in a little alcohol and neutralized with 4 cc. normal soda solution. This volume of soda solution is equivalent to 4×50 mgms. calcium carbonate and is diluted to 200 cc., enough alcohol being added that the per cent. in the liquid may be 35. The solution thus prepared gives constant results. Instead of pure palmitic acid, fatty acids separated from tallow may be used, by neutralizing, using phenol-phthalein as an indicator

and diluting according to the number of cc. of normal alkali solution used. A. H. W.

On the Nature of the Reaction Between Solutions of Ferric Chloride and Potassium Iodide and the Volumetric Titration of Iron in the Ferric State.—D. J. Carnegie, Chem. News **60**, 87. The author describes a series of experiments, undertaken to determine the conditions of the best use of potassium iodide as a reagent for volumetric analysis. The result he arrives at incline him to support the reaction of Duflos, who was the first to point out the applicability of potassium iodide for the determination of iron in the ferric state, viz.:

$FeCl_3+3KI=FeI_2+I+3KCl$, which may be divided into two stages:

(*a*) $FeCl_3+3KI=FeI_3+3KCl$.

(*b*) $FeI_3=FeI_2+I$.

The ordinary reaction given in the text-books, $FeCl_3+KI=FeCl_2+KCl+I$, he does not consider probable. A. H. W.

Note on the Analysis of Concentrated Superphosphates.—John Hughes, Chem. News **60**, 91. The fact is pointed out that concentrated superphosphate, containing from 37 to 41 per cent. of phosphoric acid soluble in water, usually has in addition from 4 to 5 per cent. of the phosphoric acid, combined with ferric oxide and alumina in the form of precipitated phosphate, which latter is not soluble in pure water, but is soluble in a concentrated solution of superphosphates. In analyzing such superphosphates, it is very important that sufficient water be added for the first extraction, otherwise the soluble phosphoric acid is returned from 1 to 2 per cent. too high. The author's method is to take 1½ gms. of the superphosphate, place in a small beaker and add a little water to soften the mass and permit the breaking up of all lumps. The volume of the solution is then increased to 90 cc. with cold water, allowed to stand five minutes, when the supernatant liquid is filtered off. To the residue, 90 cc. of water are added, the whole then carefully stirred, allowed to settle and filtered. The residue from this filtrate is then treated with 60 cc. of water, warmed gently, and the whole brought upon the filter and washed once with hot water. A. H. W.

A New Process for the Volumetric Determination of Silver, Mercury, and Thallium by Means of Potassium Iodide.—Adolphe Carnot, Chem News **60**, [illegible] Comptes Rendus **109**, 177. The method is based on the insolubility of silver and mercury iodides in a nitric acid solution when alkaline iodides are absent. The process for mercury, which must be in the mercuric state, is as follows. The solution, diluted with water to [illegible] or 150 cc., is put into a beaker with about 10 cc. of nitric acid (slightly nitrous is preferable) and a little clear starch paste and titrated with a decinormal solution of potassium iodide ([illegible] gms. per liter), the starch being used as an indicator of the end of the operation. Care must be taken not to overstep the limit when a permanent change of color to a general brownish tint takes place, otherwise the liquid, when left to itself will remain of a blue color after the subsidence of the red precipitate. The method is not applicable in the presence of free hydrochloric acid or alkaline chlorides. In the case of silver, when the substance in question is of unknown composition, an approximate analysis is made. Then the approximate weight of the silver in the amount taken for analysis is calculated. The substance is then dissolved in nitric acid, diluted to 100 cc., and the calculated amount of decinormal potassium iodide is then added, the liquid being kept briskly agitated to collect the silver iodide in the form of dense clots. Then it is decanted into a beaker and starch paste and 10 cc. of nitric acid slightly nitrous added. If at this stage the liquid becomes blue, it is exactly decolorized by a centinormal solution of silver nitrate (1.7 gms. per liter). If it is not colored, potassium iodide is added until the permanent blue tint is reached, which is then destroyed by the centinormal silver solution. Thallium may be determined though with a little less accuracy by this method, the yellow iodide being a little less insoluble than those of silver and mercury. The method is not applicable to palladium, as its iodide is of so deep a color it is impossible to tell when the free iodine begins to color the starch.

[illegible]

Valuation of Hypophosphites. Frank Moerk, Chem. Ind. Rep. **13**, [illegible]

[illegible] of the hypophosphite is dissolved in 100 cc. of

water, and bromine is added drop by drop until the color ceases to change after slightly heating.

$$NaH_2PO_2+2Br_2+2H_2O=NaH_2PO_4+4HBr$$

The excess of bromine is then driven off by boiling and 15 cc. of a neutral 10 per cent. calcium chloride solution are added, together with a few drops of phenol-phthalein solution, and the whole is titrated with standard normal caustic soda solution. The number of cc. used are multiplied by the following factors : for

$Ca(H_2PO_2)_2$	0.014166
NaH_2PO_2	0.014666
KH_2PO_2	0.01685
$Fe_2(H_2PO_2)_6$	0.01394

In case the hypophosphite contains free acid or alkali, these must be estimated and brought into the calculation. The author claims as accurate results as with the mercuric chloride method.

S. C.

Nitro Prusside of Sodium as a Reagent for the Caustic Alkalies and Alkaline Earths.—H. Brunner, Chem. Zeit. Rep. **13**, 221, finds that nitro prusside of sodium, when added to a solution of caustic alkali or alkaline earth, produces an intense yellow color, while it exerts no influence upon the carbonates. If this reagent be added to a solution of sodium hydrate until an orange color is produced and then alcohol be added, and the solution, after stirring, allowed to stand, a dark yellow oil separates out, which finally solidifies in orange colored crystals. This compound, whose formula the author has not worked out, gives colored precipitates with metallic salts and a green color with the acids. When exposed in the air some time, it changes gradually to a blue color.

S. C.

The Analysis of Glasses.—David Lindo, Chem. News **60**, 14, 33, and 41. The author, whose death occurred at Falmouth, Jamaica, May 7, had undertaken, a few months before his decease, analyses of different glasses used in the laboratory, as he found most all his beakers, etc., became cloudy. For the analysis of silica, the finely powdered glass was first ignited gently in a platinum crucible, then placed in a desiccator, carried to the balance while warm and 2 gms. were weighed out. The glass, after being

thoroughly fused in a large platinum crucible with alkaline mixture (13 parts potassium carbonate and 10 sodium carbonate) was cooled, generally by placing crucible in water. Distilled water was next added and the crucible was heated on a sand bath, when the contents were generally nearly dissolved out, and then transferred to a beaker. The cover of the crucible was finally rinsed with the hydrochloric acid (30 cc.) used in the decomposition. The next day, after heating on a sand bath, the whole was transferred to a large platinum dish and evaporated to dryness, reduced to a powder in an agate mortar and reheated on sand bath for two hours. It was then moistened with 6 or 8 cc. hydrochloric acid and a little water, allowed to stand for several hours, then 60 cc. water were added and the whole was heated and filtered into a 200 cc. flask, and washed well with hot water. The silica was dried in the stove, ignited, refused with 6 or 7 gms. alkaline mixture and the above process repeated, the solutions always being brilliantly clear by this method. To get the silica in the vitreous form requires sufficient dilution before adding the 25 cc. hydrochloric acid. 50 instead of 60 cc. of water were used the second time and the silica was collected on a Gooch crucible, and filtrate and washings added to contents of the flask. A little silica passed into solution, it was recovered by adding 10 cc. of ferric chloride, precipitating with ammonia, filtering through Gooch crucible, igniting and weighing. The silica was obtained in three parts (1) from the whole mass of salts, (called the bulk), (2) from the ammonium precipitates, (3) from the filtrate given by ammonia, carrying down the last trace with ferric oxide.

THE AMMONIA PRECIPITATE.

The filtrates and washings from the silica being all united in a 200 cc. flask, most of the liquid was poured into a beaker of hard Bohemian glass, and concentrated. Ammonia was added until a permanent precipitate formed, which was then redissolved by adding the remainder of the acid liquid in the flask. The whole was now transferred to flask, and sulphuretted hydrogen was passed through for fifteen or twenty minutes. It was then kept in a closed flask for two or three days, filtered through Gooch crucible, transferred to a beaker, and concentrated. A few drops of nitric acid were added, the solution was heated and precipitated

with ammonia. The filtrate and washings were collected in a 200 cc. flask, ammonia and a little ammonium sulphide were added, and the contents diluted to the mark when cold. The ammonia precipitate was treated in one of four ways.

First method.—Separated silica by igniting strongly, fusing with a little potassium bisulphate, boiling out with hydrochloric acid and water, and evaporating to dryness. 1 gm. of citric acid, ammonia and acetic acid were added to filtrate from silica, and lime was tested for with oxalate. Magnesia mixture and ammonia were used for separating phosphoric acid if present, and the iron was precipitated as sulphide by ammonium sulphide and weighed after oxidation, and precipitated by ammonia as ferric oxide. The alumina was found by difference.

Second method.—Silica was separated after fusion with alkaline mixture, ammonia and ammonium oxalate were added to filtrate, to precipitate any lime. 1 gm. of citric acid, ammonia in excess, and 30 cc. of magnesia mixture were then added to filtrate from lime, if present, and iron and alumina were found as in first method.

Third method.—When phosphoric acid was not present in appreciable quantity, the precipitate was not ignited, simply dried, and total weight was not taken as was done in first two methods. After silica was separated, the concentrated filtrate was added to a solution of soda, and the ferric oxide collected on a Gooch crucible. Alumina is precipitated in filtrate by ammonia.

Fourth method.—Dissolved the precipitate in hot hydrochloric acid on filter. If any silica seemed to be retained by filter, the paper was burnt, and ash was added to filtrate. After separation of silica, iron and alumina were determined as in preceding method.

THE LIME AND MAGNESIA.

After precipitating manganese in the 200 cc. flask with ammonium sulphide, 100 cc. = 1 gm. of filtrate were taken for the determination of lime and magnesia. Hydrochloric acid was added in slight excess and the liquid was concentrated and filtered and lime was precipitated by ammonium oxalate. If more than traces of magnesia were found in filtrate, tested for by microcosmic salt, the precipitate of calcium oxalate was dissolved by hydrochloric

acid, reprecipitated, filtered, and filtrate added to liquid in which the bulk of magnesia is precipitated. 50 cc. of the 200 cc. solution were filtered, acidulated with hydrochloric acid, concentrated, filtered, 10 cc. of ferric chloride were added and ammonia was used to precipitate the iron to recover lost traces of silica. A little silica may be carried down with the lime, so when but moderate quantities of lime were present, the author did not remove this before recovering silica. Interesting results of the analyses are embodied in the articles. A. H. W.

The Ammonia-Cobaltic Molybdates, Tungstates, and Vanadates. Separation of Nickel and Cobalt and Cobaltous and Cobaltic Salts.—Adolphe Carnot, Chem. News **60,** 54 (Comptes Rendus **109,** 109). The author records his investigations with ammonia-cobaltic molybdates only. If ammonium molybdate be added to a solution of cobalt which has been first converted into the purpurco-cobaltic salt by hydrogen dioxide, in presence of ammonium chloride and ammonia (the author's method—Comptes Rendus, April 8, 1889, this Journal **3,** 324), there appears no turbidity. But if the solution be neutralized with acetic acid, a bulky precipitate of a fine peach blossom color is produced, which is almost insoluble. Filtered, washed, and dried at 100°, it retains a rose color, but heated more strongly, takes successively violet, blackish green and greenish yellow colors, corresponding to eliminations of water, ammonia and oxygen. The matter is converted into cobaltous molybdate and has a lilac color when cold. If dull redness is exceeded, it suffers a partial dissociation, and black cobalt oxide and molybdic anhydride in small crystals are formed. The composition of the dried salt is given as Co_2O_3, $5NH_3$, $7MoO_3+3H_2O$, and calcined, $2CoO_7$, $7MoO_3$. The analysis consisted in decomposing the rose-colored salt (after being dried at 100°) with potash lye, collecting the ammonia in a standard solution of sulphuric acid, isolating the insoluble cobalt peroxide and determining the cobalt after reduction by hydrogen. The alkaline molybdate was converted into sulpho-molybdate and the molybdenum weighed as sulphide. The water was found by difference.

The salts of cobaltous oxide and the nickel salts form no precipitate with ammonium molybdate either in an acetic or ammoniacal

solution. Hence a ready method of separating the ammonia cobaltic from the ammonia-cobaltous salts and also of separating nickel and cobalt. The details of the latter separation are as follows: The two metals in hydrochloric or nitric solution are treated with hydrogen dioxide and the cobalt is thus peroxidized. The solution is afterward neutralized with acetic acid, and excess of ammonium molybdate is added until there is no further turbidity. After washing by decantation and drying, it is separated from the filter paper which is burnt separately. The precipitate is finally ignited at a temperature not exceeding dull redness, cooled, and weighed. The weight multiplied by the coefficient 0.1332 equals cobaltous oxide and by 0.1048 gives the weight of the metal. This colored precipitate of ammonia cobalt molybdate affords also a trustworthy qualitative test for cobalt. The nickel is determined in the filtrate from the precipitate of the cobaltic salt, by precipitating with caustic potash, boiling to expel ammonia, filtering, redissolving in hydrochloric or nitric acid, and reprecipitating with potassium hydroxide and bromine. The sesquioxide of nickel contains no trace of molybdate and is determined either by means of a standard solution or by reducing in hydrogen and weighing the metallic nickel. A. H. W.

Precipitation of Manganese as Peroxide.—Herman Alt, Chem. Zeit. **13,** 1339. The method, in which manganous salts are converted into manganic salts by a halogen and then precipitated by ammonia, is troublesome on account of the precipitate adhering to the flask. The author overcomes this difficulty by heating the slightly acid manganous solution in a flask until free from air and then adding ammonium chloride and making alkaline with ammonium hydroxide. This produces no turbidity. The solution is then heated to boiling and bromine vapor is drawn through with a filter pump, which causes the peroxide to separate quickly, and may easily be brought on the filter.

S. C.

Nitrites in Water.—According to C. Wurster, Ber. **22,** 1909, an addition of ammonium acetate to the water to be tested for nitrites according to Griess' methods (Ber. **11,** 624; **12,** 427) causes a much more rapid development of the color. To 10 cc. of the liquid to be tested the author adds ½ cc. of ammonium acetate

solution made by adding 4 parts glacial acetic acid to 3 parts ammonia. For detecting nitrites the author recommends the use of the yellow or red Griess' reagent papers sold by Schuchardt of Görlitz. The yellow paper is impregnated with metaphenylene diamine and the red with alpha naphtylamine and sulphanilic acid.

Alpha Naphtylamine as a Reagent for Hydrogen Dioxide in Presence of Common Salt.—C. Wurster, Ber. **22**, 1910. When dilute solutions of hydrogen dioxide and naphtylamine are brought together in acetic acid solution no action takes place, but if common salt be added oxidation takes place and naphtameine is formed, giving the solution a blue or violet blue color.

Spectro-colorimetric Determination of Iron and of Thiocyanates.—Krüss and Moraht, Ber **22**, 2054. When ferric salts and thiocyanates are mixed, the maximum of absorption is not obtained until for every molecule of the ferric salt 12 molecules of the thiocyanate have been added. The colored salt produced has the composition, if potassium thiocyanate be used, $Fe(CNS)_3$. $9KCNS$. The concentration of the solution and the presence of other salts and of acids, has an influence upon the absorption spectrum, so that this method cannot well be used for the determination of iron and thiocyanates.

Determination of the Amount of Lime and Soda to be added in the Purification of Water.—O Binder, Ztschr anal Chem **27**, 176. The amount of lime or soda to be added to a water for its purification may be determined from the analyses but when it is not convenient to make a complete analyses this may be determined as follows.

I. DETERMINATION OF THE AMOUNT OF LIME.

To 200 cc water in a 300 cc flask, 50–75 cc saturated lime water whose content in lime is exactly known, is added. The lime water is titrated against sulphuric acid made by diluting 46.43 cc normal acid to a liter. The flask is heated to the temperature at which the purification on the large scale is to be conducted, allowed to cool, filled to the mark with distilled water free from carbonic acid, 200 cc filtered through a dry filter and

the excess of lime in the filtrate determined with the standardized sulphuric acid, using phenol-phtalein as the indicator.

II. DETERMINATION OF THE AMOUNT OF SODA.

250–300 cc. of water is evaporated to dryness in a platinum dish, the residue dissolved in water, the solution filtered, and the excess of soda in the filtrate determined by means of acid and methyl orange. To the necessary quantity of soda thus found it is necessary to add about 10 gms. per cubic meter of water, even for waters which tested as above require no soda.

The Determination of Small Amounts of Arsenic in Woven and Spun Goods, etc.—R. Fresenius and E. Hintz, Ztschr. anal. Chem. **27,** 179. 25 gms. of the substance are placed in a half liter retort and ¼ liter concentrated hydrochloric acid added. The neck of the retort was bent slightly, the end being connected with a condenser. The neck of the retort next the tubulure pointed upwards so that any material carried into it mechanically would return to the retort. The end of the condenser tube was connected with a tubulated receiver containing 200 cc. water and placed in cold water, and the tubulure was tightly closed with a cork, through which passed the end of a Peligot's tube containing water.

After digesting the mixture about an hour, 5 cc. of a concentrated ferrous chloride solution was added, the retort stopper luted with vaseline and firmly closed, and the solution distilled until the residue frothed so strongly as to put a stop to it. In this way two-thirds of the contents of the retort can be distilled over. After cooling, 100 cc. hydrochloric acid is added, and the distillation repeated. The distillate, which has more or less color, is treated with sulphuretted hydrogen, at first warm, and then allowed to cool and stand for 12 hours. The impure sulphide is filtered through asbestos in a funnel which has a stop-cock in the neck. After it is washed, the stop-cock is closed, and the precipitate covered with strong hydrochloric acid in which bromine has been dissolved. The solution is then run into the flask in which the precipitation was made, and the solution brought again into the retort, and the distillation repeated. Flask and funnel are washed with concentrated hydrochloric acid. After the second distilla-

tion, the arsenic is precipitated as sulphide, washed with water, alcohol, carbon disulphide and alcohol, dried at 110°, and weighed. This method may also be used to determine the arsenic in colors, as in fuchsine.

The Cyanide Method of Estimating Copper.—G. E. R. Ellis, J. Soc. Chem. Ind. **8,** 686. The author gives a series of results obtained with this method upon known amounts of copper in solution with varying amounts of zinc, and comes to the following conclusions :

"From these results it may be concluded that the presence of 4–5 per cent. of zinc in the copper solution does not practically affect the correctness of the indications afforded by the potassium cyanide solution ; but that when the percentage of zinc exceeds that amount, the indications of the standard solution are perfectly untrustworthy, and that consequently the zinc must be removed before titration is effected. When the percentage of zinc present was considerable, it was noticed that the change of color of the solution from light blue to faint violet took place very slowly indeed.

It seemed also a matter of interest to determine whether the "copper value" of the blue solution altered on being kept exposed to air and to light for a considerable time. All the experiments performed showed that the blue color in the ammoniacal solution at the end of three weeks required for its discharge practically the same volume of potassium cyanide solution as it did when first produced. For instance :

On June 1st a solution (A.) was prepared containing 20 cc. of solution of copper nitrate, 100 cc. of water, and 10 cc. of ammonia solution. The blue solution, which did not fill the flask, was then exposed to light until the 22nd June, when it was titrated and required 17.5 cc. of cyanide solution for the discharge of the color.

Solution (B.), prepared and titrated on the 22nd with the same solutions, containing 30 cc. of copper nitrate solution, 150 cc. of water, and 15 cc. of ammonia solution, required 26.3 cc. of cyanide solution.

$$26.3 \text{ cc. for } 30 = 17.53 \text{ cc. for } 20.$$

As regards the standardising of the potassium cyanide solution, my experience seems to indicate that standardising should be performed *at least* once a week ; a certain weight of copper which on the 6th of June required 24.8 cc. of cyanide solution needed 26.3 cc. of the same solution on the 22nd of the same month—an increase of 0.1 cc. per day.

I hope soon to be able to present to the Society a series of determinations upon the influence of varying quantities of iron on the amount of potassium cyanide required for the titration of solutions containing equal weights of copper. Present results show that the percentages obtained are always low when a considerable percentage of iron is present, owing probably to the retention by the precipitated hydrate of a portion of the blue ammoniacal compound.''

Copper Sulphate as an Indicator for the Titration of Zinc and Manganese.—F. Moldenhauer, Chem. Zeit. **18,** 1220. In this method an ammoniacal solution of zinc is titrated with a $\frac{n}{10}$ solution of potassium ferrocyanide.

The indicator is prepared by saturating strips of filter paper for half their length with a 4 per cent. solution of copper sulphate and quickly dried. They are then cut into strips from 2 to 4 mm. in width, and are ready for use. A drop of the solution to be tested is placed on the opposite end of the paper, and it unites with the copper sulphate solution by capillary attraction. If a small excess of the potassium ferrocyanide solution be present, a red line is produced at the junction of the two solutions in the test paper. If only a trace of the above salt be present in excess, the red line does not appear immediately, but very soon after. An excess of 0.1–0.2 cc. of $\frac{n}{10}$ solution in 100 cc. ammoniacal water gives a distinct reaction. Freshly made papers are more sensitive than old ones, but the latter may be made sensitive by moistening with acetic acid vapor.

The solution to be titrated gives more satisfactory results if warm, because it acts more rapidly in the filter paper, and the ammonia evaporates so that it does not influence the test.

If manganese is present, a sulphuric acid solution is used, by which the lead (which under no consideration should be in the ammoniacal zinc solution) is removed, and the filtrate is made alkaline with ammonia. The precipitate formed is filtered off,

washed, redissolved in sulphuric acid, and again precipitated with ammonia. The united filtrates are diluted to a known bulk, and part of the solution is titrated with the ferrocyanide solution before it becomes turbid through higher oxidation. Another part of the filtrate is acidified with sulphuric acid, and some zinc oxide is added. The nearly neutral solution is then heated to boiling, and the manganese determined by Volhard's method with potassium permanganate. The number of cubic centimeters of ferrocyanide solution corresponding to the number of permanganate solution used (found by previous standardization) is subtracted from the number obtained by the first titration, and the result will give the amount of zinc.

If zinc is absent, manganese may be determined by titrating the slightly acid or neutral manganous solution with $\frac{n}{10}$ potassium ferrocyanide solution. It must be remembered that in these cases the manganese replaces only one equivalent of hydrogen, while zinc replaces two in the potassium ferrocyanide. S. C.

Electrolytic Determination of Antimony.—Ad. Lecrenier (Chem. Zeit. **18,** 1219) gives the following modification of Classen's method of precipitating antimony from a solution of the sulphide. The antimony solution, no matter what the degree of oxidation may be, is treated with an excess of sodium sulphide and 50–75 cc. of a 20 per cent. solution of crystaline sodium sulphite are added. The solution is then carefully heated until colorless, and, after allowing to cool, is attached to the battery. A current liberating 2.5 cc OH gas a minute is sufficient.

The antimony adheres firmly to the dish, and is free from sulphur. Not more than 0.2 gm. of antimony should be deposited. Notwithstanding the excess of sodium sulphite used, a little sulphur is deposited on the positive electrode, but it does not affect the result. S. C.

Precipitation of Copper as Sulphocyanide.—F. Johnson, J. Soc. Chem. Ind. **8,** 603. Dissolve the ore in any suitable acid, boil off the excess of acid, dilute with about twenty times the bulk of water, and add three times the weight of copper present of ammonium sulphocyanide. This will, with ferric salts, produce a deep red solution, to which add stannous chloride until bleaching ensues. A liquid containing cuprous sulphocyanide

and the insoluble portion of the ore results. Filter and wash with water containing about 1 per cent. hydrochloric acid. Drop the precipitate and paper into a dissolving flask and pour on 50 cc. of a dilute acid mixture of 1 part nitric acid, 1 hydrochloric, and 4 water. Boil gently until all red fumes are given off, and then evaporate to one-fourth bulk. Add ammonium hydroxide, and when fairly cool, titrate with a solution of potassium cyanide. The solution of potassium cyanide is standardized preferably with copper dissolved in acid, and precipitated as sulphocyanide, and redissolved in ammonia.

Another method.—Drop the paper containing the precipitate of cuprous sulphocyanide and insoluble portions of the ore into the dissolving flask as above, and pour 50 cc. acid ferric sulphate solution over it. This solution contains about 100 gms. ferric sulphate and 50 cc. H_2SO_4 per liter. Mix well and run in permanganate solution until brown or olive green. Simmer 5 or 10 minutes, add 250 cc. water, and complete permanganate titration. A good strength of the permanganate solution is 20 gms. per liter.

O. O. L.

IRON AND STEEL.

EDITED BY P. W. SHIMER.

The Desulphurization of Pyritiferous Iron-Ores.—Sterling G. Valentine, Ph. D. (Trans. A. I. M. E., Colorado Meeting, June, 1889). The author gives the details of a series of experiments made to determine the proper conditions for the elimination of sulphur from ores when present as FeS_2. The following conclusions are drawn from the investigation :

1. Heat alone, without access of air, can remove, at best, only one-half of the sulphur present.

2. Atmospheric oxygen is absolutely necessary for a proper desulphurization.

3. Even at a low heat, ore is properly desulphurized if air can gain access freely to the FeS_2 in it.

4. Sulphate of iron can be decomposed by heat equally well with or without air.

5. In order that the residue of sulphur in roasted ores may consist, so far as possible, of sulphates, the roasting must be done under free access of air.

6. Sintering does not allow much of the remaining sulphur to be in the form of sulphate.

7. Fusion, hence, should never occur in roasting, except after continued heating in air at a low temperature.

8. Ores cannot be properly desulphurized in the upper part of the blast-furnace.

9. An efficient roaster must allow easy control of heat, abundant air access to the hot ore, and rapid removal of the products of combustion.

A Rapid Method for the Determination of Phosphorus in Certain Ores.—J. Reed Woodbridge (Trans. A. I. M. E., New York meeting, June, 1889). Dilute nitric acid is used for making the solution of the ore in this method. It is applicable only to those ores in which all the phosphorus is known to be present as apatite. In the case of the magnetic iron ore, on which the experiments were principally made, the results obtained agree closely with those obtained by the standard methods.

MINERAL ANALYSIS.

EDITED BY JOHN EYERMAN.

Gadolinite from Texas.—F. A. Genth (Am. J. Sci., Vol. 38, Sept., 1889, p. 198) gives analyses of this mineral from Burnett (*a*) and Llano (*b*) Counties, Texas.

	a		*b*	
SiO_2	22.87†	23.40	22.80	22.92
Al_2O_3	0.28	0.33	0.31	0.29
Ce_2O_3*	2.65	2.76	2.66	2.85
$(Di\ La)_2O_3$	5.22	5.17	5.01	5.33
$(Y.\ Er)_2O_3$	44.35	44.65	44.45	44.30
MnO	0.22	not det.	0.18	not det.
FeO	13.69†	13.58	12.93	13.03
BeO	9.24	9.32	9.19	9.34
MgO	0.07	0.08	0.11	not det.
CaO	0.64	0.54	0.71	0.78
Na_2O	0.20	not det.	0.23	not det.
K_2O	0.15	not det.	0.12	not det.
Ignition	0.72	not det.	0.79	not det.
Insol. in dil. H_2SO_4		not det.	0.93	0.92
	100.30		100.42	

*Contains ThO_2.
†Includes the Fe_2O_3 and SiO_2, insoluble in dilute Sulphuric acid.

Monazite from Canada.—F. A. Genth (*Ibid* p. 203) has analysed an interesting variety of this rare mineral, lately discovered at the Villeneuve Mica mine, Ottawa Co., Quebec. Color, reddish-brown. Sp. gr. 5.233.

$(LaDi)_2O_3$	$(Y\ Er.)_2O_3$	H_2O	SiO_2	ThO_2	P_2O_5	Fe_2O_3	Ce_2O_3	MgO	CaO
26.41	4.76	0.78	0.91	12.60	26.86	1.07	24.80	0.04	1.54=99.77

Two Sulphantimonites from Colorado.—L. G. Eakins (Proc. Col. Sci. Soc. III, 1888, p. 73) describes and gives analyses of these interesting minerals, from Gunnison Co., Col.

a. "Domingo" Mine.

b. Augusta Mountain, one mile east of locality *a*.

	Ag	Cu	Pb	Fe	Mn	Sb	S	Insol.
a	Tr.	Tr.	39.33	1.77	Tr.	36.34	21.19	.52 = 99.15
b	Tr.	Tr.	55.52	Tr.		25.99	18.98	=100.49

Formula *a* $(Pb\ Fe)_3\ Sb_4S_9$, or $3(Pb\ Fe)\ S2Sb_2S_3$

b $Pb_5Sb_4S_{11}$, or $5PbS,\ 2Sb_2S_3$

Kobellite, from Ouray, Col.—H. F. Keller (Jour. Frank. Inst., Aug., 1889) in an exhaustive paper gives the composition, chemical properties, etc., of this mineral from the Silver Bell mine, Ouray, Col.

After deducting impurities (chalcopyrite, gangue) he calculated the following results:

S	17.76	.5550		=	2.600
Bi	30.61	.1457	.2134	=	1.000
Sb	8.13	.0677			
Pb	38.95	.1881	.2124	=	.995
Ag	3.58	.0166			
Cu	.97	.0077			
	100.00				

Formula: $2(Pb,\ Ag_2Cu_2)S.(BiSb)_2S_3$

Sp. gr., 6.334. H. 2.5–3.

Plattnerite from Idaho.—H. A. Wheeler (Am. J. Sci. 38, p. 79) communicates a short description and an analysis of this plumbic oxide from the Cœur d' Alene District, Idaho.

Color, iron black; streak, chestnut-brown; hardness, 5–5.5. Sp. gr. 9.411; fusibility 2. Soluble in hydrochloric acid and aqua-regia.

Pb	83.69%

PbO_2	96.63
SiO_2	1.62
Fe_2O_3	1.12
	99.37

Iron Sulphates from Chili.—G. Linck (Zeit. f. Kryst. XV, p. 1) gives an exhaustive description and a number of analyses of iron sulphates found near Copiapo, Chili:

a	Coquimbite	Sp. gr.	2.105
b	Quenstedtite	Sp. gr.	2.1155
c	Copiapite	Sp. gr.	2.103
d	Stypticite	Sp. gr.	1.857
e	Romerite	Sp. gr.	2.102

	a	*b*	*c*	*d*	*e*
SiO_2	1.29			FeO	[illegible]
SO_3	41.48	39.81	38.91	32.94	38.47
Fe_2O_3	27.56	27.66	30.10	32.41	17.63
Al_2O_3	trace	trace	trace	trace	[illegible]
MgO	trace	trace		trace	
H_2O	28.77	31.55	30.74	34.32	[illegible]
CaO		[illegible]	trace	[illegible]	trace
Insoluble				[illegible]	
	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

Dudgeonite, a New Mineral.—M. Forster Heddle (Min. Mag. May, [illegible], p. [illegible]) describes and gives analysis of this new mineral from near Creetown, Kirkcudbrightshire, Eng. H. = [illegible]. Fracture earthy. Streak lustrous or waxy.

NiO	[illegible]
CoO	[illegible]
CaO	[illegible]
As_2O_5	[illegible]
H_2O	[illegible]
	[illegible]

Formula [illegible] NiO [illegible] CaO As_2O_5 [illegible] H_2O. Named after its discoverer Mr. Dudgeon.

ORGANIC ANALYSIS.

Estimation of the Fats in Milk.—Th. Dietrich, Ztschr. angew. Chem. 1889, 413.

The author outlines the method used at the Marburg experiment station. J. E. W.

Detection and Determination of Sodium Bicarbonate in Milk.—L. Padé, Chem. News **60,** 81 (Comptes Rendus **109,** 154).

Sodium bicarbonate is the salt most commonly added to milk to prevent coagulation, but as, during ignition, about two-thirds of the carbonate is converted into sodium phosphate and calcium carbonate by reacting upon the calcium phosphate of the milk, the author has devised the following method: 25 cc. of the milk are incinerated and the alkalinity of the ash determined by decinormal sulphuric acid. The volume, obtained in cc., multiplied by 0.0084 gms., gives the quantity of bicarbonate, not converted into sodium phosphate, contained in 25 cc. of milk, and this is afterwards multiplied by 0.0336 to obtain the percentage. In this neutral solution, mixed with 2 cc. of sodium acetate at 10 per cent., slightly acidulated with acetic acid, the phosphoric acid is determined by a standard uranium acetate solution, so standardized that 1 cc. equals 0.01 gms. of sodium bicarbonate in 100 cc. of milk, when 25 cc. are taken for analysis. The reaction on which the method is based is

$$4(HNaCO_3)+Ca_32PO_4=2HNa_2PO_4+3CaCO_3+CO_2+H_2O.$$

The uranium solution is standardized with a solution containing 3.11 gms. sodium ammonium phosphate per liter, this weight of sodium phosphate being equivalent to 2.5 gms. sodium bicarbonate and if the determination is made upon 25 cc. of milk, 1cc. of the standard solution of uranium,or of sodium phosphate corresponds to 0.01 gms. bicarbonate in 100 cc. of milk. Either ferrocyanide or tincture of cochineal are used as indicators, the latter being the more sensitive. The weight per cent. of alkalinity added to that obtained by the determination of the phosphoric acid, gives the total per cent. of bicarbonate added to the milk. A. H. W.

The Estimation of the Sugars, and a Polarimetric Ex-

amination of Sweet Wines.—A. Borntrager, Ztschr. angew. Chem. 1889, 477.

The author describes his methods of treating different kinds of wine, and gives tables comparing the results obtained by his analyses and observations. J. E. W.

Starch Determination.—A. von Asboth (Chem. Zeit. **13**, 591) finds that Lintner's method gives low results when used with anything but pure starch, while the baryta method is applicable to all cases and gives good results. To make the latter method universal, it is necessary to extract all the fats or fatty acids from the grain, and to do this, he uses ether in Soxhlet's apparatus. The remainder of the analysis is unchanged. S. C.

New Method for Determining Glycerine in Wine and Beer. H. Graf von Touring, Ztschr. angew. Chem. 1889, [illegible]. A convenient quantity of the material (Wein's Beer 50 cc. [illegible]) is evaporated to 10 cc. on the water bath, 15 gms. of burnt gypsum added, and extracted with absolute alcohol for six hours. To the extract is added 15 cc. to 25 cc. of water, and it is then distilled under diminished pressure.

The apparatus for distilling consists of an ordinary glass retort connected with a small condenser, the lower end of which passes into an Erlenmeyer flask connected with a suction pump. The retort is heated in a sheet iron air bath at from 150° to 1[illegible] until all the water is expelled, then the pump is attached and the temperature raised to 19[illegible]–210°.

The last traces of glycerine are washed from the retort and condensed by distilling 3 cc. or 4 cc. of water at ordinary pressure. The glycerine in the distillate is then determined as benzoate by the method of Diez. J. D. W.

A Method for the Detection of Chlorine, Bromine, Iodine and Sulphur in Organic Compounds. Charles W. Marsh (Amer. Chem. Journ. 1889, **11**, 24[illegible]).

The author recommends heating the organic substance supposed to contain one of the above mentioned elements with metallic zinc in the form of powder in a closed tube until the substance is decomposed by the heat.

The tube and contents are cooled. The test for sulphur is made by adding a few drops of dilute hydrochloric acid and lead acetate paper, and by the odor.

Chlorine is detected by adding to the cooled contents of the tube about one cubic centimeter of water, shaking and filtering. The filtrate is divided in two parts and a few drops of nitric acid added to each. In one portion chlorine is detected by means of silver nitrate as usual, and to the other carbon disulphide is added. The carbon disulphide should not be colored.

The tests for bromine and iodine are made in a similar manner, the reaction with silver nitrate giving the corresponding silver precipitates, and the brown and violet colors respectively with carbon disulphide. The zinc powder is often impure and should be previously tested by blank experiments. C. W. M.

Advantages of Kjeldahl's Method of Estimating Nitrogen.—The authors mention the following points in which the method of Kjeldahl is in their opinion superior to that of Will and Varrentrapp :

1. The state of dryness of the matter to be analyzed. Many agricultural products often have from 15 per cent. to 40 per cent. of water and their mixture with soda lime sometimes gives rise to a disengagement of ammonia, whence the necessity of thoroughly drying these substances.

2. The liquid or pasty state of some bodies, beer, milk, etc., are examples of this class.

3. The great bulk of certain light bodies and their hygroscopicity.

4. The heterogenity of certain samples.

5. The resistance which certain samples present to pulverization, for instance, horn and hair.

All these difficulties disappear in the application of Kjeldahl's method, and this process has been employed for several years in Denmark, Italy, Germany, United States and France.

Aubin and Alla, Comptes Rendus, Vol. 108, No. 18, p. 960.

H. W. W.

Estimation of Organic Nitrogen by the Volumetric, Soda-Lime and Kjeldahl Methods.—(L. l'Hote, Comptes Rendus, 108, 15th April, 1889, p. 817.

The results obtained are shown in the following table

Substance	Theory %	% Soda Lime	% Kjeldahl	Time h	Time m	Kjeldahl Color of Liquid
Brucin	7.10	7.15	5.89	16	00	Colored
Cinchonin Sulphate	7.46	7.42	3.40	1	30	
"	7.46	7.42	3.92	17	00	
Asparagin	18.66	18.42	18.33		45	Almost colorless
Caffein	28.86	28.78	28.74		"	"
Oxamide	31.82	31.68	31.67			"
Uric Acid	33.33	33.27	33.18			"
Urea	46.66	46.54	46.50			"

It is seen from the above figures that for some substances the results of the two processes are almost identical. Substances like the first three, however, are very refractory, not yielding all their nitrogen in an oxidised form even after 37 hours boiling with sulphuric acid. Blood, casein and wheat give up all their nitrogen but some of it is lost by volatilization, so that the results are too low.

In the case of casein only 7.50 per cent nitrogen are found instead of 13.35 per cent by soda-lime. The boiling continued for 13 hours.

In another sample, heated for 37 hours with Nordhausen acid, 12.90 per cent were found.

The transformation of organic into ammoniacal nitrogen by Kjeldahl's method is easy for some crystalline substances like oxamide, caffein, urea, etc.; [illegible] is not always possible even after a long time with many [illegible] products.

[illegible] Bulletin de la Soc. Chem. vol. [illegible] that the nitrogen in urine can not be exactly de-

termined by the soda-lime method. It contains some albuminoid compounds which are not completely transformed by the soda-lime. They further say, that it has long been known that it is impossible to estimate the nitrogen in albumen by the soda-lime method. L'Hote has repeated the experiments of Messrs. Cazeneuve and Hugouneng on urine and albumen. With urine the results obtained by oxide of copper and soda-lime, were as follows:

	Oxide of Copper.	*Soda-Lime.*
Urine	8.86	8.72 (per liter)
Albumen	14.49	14.38 (per hundred)

These figures show that the two methods give essentially the same results. H. W. W.

On the Analysis of Cinchonas, and on the Relative Solubility of Their Proximate Principles in Water, Alcohol and Dilute Hydrochloric Acid.—Ed. Landrin, Chem. News **59**, 196 (Comptes Rendus **108**, 750). The bark of the cinchona succirubra, from the plantation of the Dutch government in Java was selected for analysis. 300 gms. of bark were taken and reduced to a powder fine enough to pass through a No. 40 sieve. The method of procedure is as follows: A milk of lime is prepared, made by adding to 75 gms. caustic lime, 75 gms. of caustic soda solution at 40° B. and 1 liter of water. This mixture of lime and soda is very necessary for liberating the total alkaloids. The bark is poured into this mixture so as to be homogeneous, and a little water may be added if it is too thick. Two liters of shale oil are then poured on the mass, and the temperature is then raised to 100° for twenty minutes, with a constant agitation of the contents. The oil is then decanted off, two more liters are added and the operation repeated as before, when all of the alkaloids of the bark will be in solution in the four liters of oil. The hot oil is then washed, at first with 75 cc. of water acidulated with sulphuric acid at one-tenth, to which 150 cc. of water have been added. This washing lasts ten minutes and the acid liquid is then poured off, the operation being repeated a second time with the same quantity of the same liquid, but the third time only half as much is employed. The first two acid liquids are united, neutralized with ammonia at a boiling heat to separate resinous matter, the same process being also used for the third acid liquid,

which serves to wash the filter of the first operation. On cooling, about nine-tenths of the alkaloid salts crystallize out. These sulphates are separated by filtration and the mother liquors are precipitated with caustic soda. The alkaloids are drained, dried and pressed, taken up in a minimum of acidulated water, so as to transform them into sulphates without acidifying the liquid. The total sulphates are then weighed and separated according to known methods. The author finds that water extracts from cinchona bark the chief part of its active principles. Alcohol at [illegible] extracts approximately the same quantity of alkaloids as water, but hydrochloric acid extracts less. A. H. W.

Examination of Commercial Olein for Linoleic Acid.

K. Hazura, Ztschr. angew. Chem. 1889, 283.

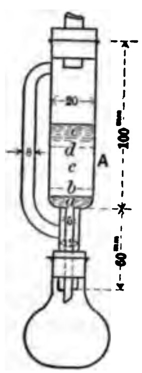
50 gms. of the olein in question is saponified, on the water bath with a very dilute alcoholic potash solution. The potash soap then obtained is freed from alcohol and dissolved in one liter of water. The solution, which should be strongly alkaline, is then mixed by slowly stirring, with one liter of a 5 per cent. solution of potassium permanganate. After one half to one hour the precipitate of hydrated manganese peroxide is filtered off, the filtrate acidulated with sulphuric acid and again filtered; this filtrate is neutralized with caustic potash and evaporated to 300 cc., then acidulated with sulphuric acid, which causes another precipitate to form. The acid solution without separating the precipitate is shaken with ether. If the precipitate completely dissolves in the ether the olein is in this case entirely free from linoleic acid. If the precipitate does not dissolve one can conclude that linoleic acid is present. To verify this, filter off the precipitate, crystallize from water or alcohol with the addition of animal charcoal and after air drying determine the melting point of the product. If this is above [illegible] the presence of linoleic acid is shown [illegible] doubt. [illegible]

The Valuation of Indigo. M. Honig, Ztschr. angew. Chem. 1889, [illegible]. [illegible] gms. of the pulverized indigo after sifting through bolting cloth is mixed as thoroughly as possible with three or four times its volume of pumice stone broken in pieces the size of corn.

An extraction apparatus of the form shown in the Fig. is used. A layer of cotton (*a*) is placed in the bottom of the tube *A*. On this is a thin layer of washed sand (*b*). The mixture (*c*), prepared as above, rests on the sand, and is covered by another layer of sand (*d*), the whole being capped by a layer of cotton (*e*). The whole charge should fill about two-thirds of the tube.

The extraction tube is connected at the top with a condensing tube, and at the bottom it is fixed into a boiling flask by a secure cork.

50 cc. of aniline or nitrobenzol, perfectly free from water, is placed in the flask and heated, care being taken that the apparatus is in a perpendicular position.



The condensed aniline or nitrobenzol, on passing through the mixture after one and one-half to two hours, will be clear or only slightly colored.

Allow the apparatus to cool, separate the tube *A* from the flask, wash any liquid out of the tube with 95 per cent. alcohol and add the washings to the flask.

The liquid in the flask is then distilled, leaving as a residue the indigotine which is washed with five or six times its volume of strong alcohol filtered on asbestos, and dried to constant weight at 110°. J. E. W.

Richness of Wheat in Gluten.—It is concluded from an extensive study of the influence of different kinds of fertilizers upon the amount of gluten in wheat that it is possible by judicious fertilizing to increase materially the quantity of this substance present in that cereal. It is sufficient for this purpose to be careful to apply to the soil as fertilizer, after harvests, such as the sugar beet, which exhausts the nitrogen, some nitrogenized substance, but not in sufficient quantities to produce a lodging or heating of the crop, and at the same time to sow those kinds which are at the same time productive and rich in gluten. (Gatellier & L'Hote, Comptes Rendus, Vol. 108, No. 20, p. 1064.) H. W. W.

A Volumetric Method for the Estimation of Fat in Milk.—G. E. Patrick, Chem. News **60**, 5.

This method consists in bringing all the milk solids, except

fat, into acid solution, in a solvent which is without sensible action upon the fat, and then measuring the latter at a fixed temperature in a narrow calibrated tube. The best solvent is found to be a mixture of strong acetic acid (80 per cent. or higher), oil of vitriol and concentrated hydrochloric acid in the proportion of 9, 5, and 2 by volume. The hydrochloric acid is added after the mixture of the other two has cooled. Should the milk solution be turbid, the proportion of acetic acid may be increased, this acid, although it retards the solvent action somewhat, giving a clearer solution of the milk solids. 10.8 cc. of milk and 15 cc. of the solvent are then taken for an analysis. The tube used is about 30 cm. long, about 17 mm. caliber, except for about 7 or 8 cm. in the middle where it is reduced to about 5 mm. caliber. The body of the tube holds about 21 to 22 cc., and when charged the liquid reaches about 2 cm. above the narrow part. The liquid may be gradually withdrawn and the fat made to enter the narrow part (which is graduated in divisions of .025 cc. each), by means of a lateral perforation in the body of the tube kept closed by a rubber band, (a section of tubing) also perforated. If 10.8 cc. of milk are taken, one such division equals 0.2 per cent. of fat, measured at 60° C., assuming average densities for fat and milk. In the lower part of the tube, two pieces of pumice stone joined together by a bit of platinum wire are placed before charging, to promote regular boiling. The milk is first pipetted in, half or more of the acid mixture is next added and well shaken with it, then the remainder is poured in, or enough to raise the liquid to the proper height. The tube is then placed on a sand bath, and its contents are boiled briskly for ten minutes, then gently for ten minutes, or more, in order to collect the fat in a layer at the top and keep it continually agitated with an up-and-down motion, until perfectly clear. When the fat layer is perfectly clear, the tube is at once placed in water at 60° C. for ten minutes, and may at once be lowered into the graduated tube if great accuracy is not essential, and its volume may be read at 60°, after replacing it in water for a few minutes. A little fat usually adheres to the tube in the descent, so, for exact results, the column of fat is only lowered until its top is just over the graduated tube, when the adhering fat is washed down with a few drops of ether, and the

tube is replaced in hot water until the ether is entirely expelled, when the fat is lowered and read at 60° C. A. H. W.

Invert Sugar.—Jungfleisch and Grimbert* have shown that the rotatory power of pure levulose, when observed directly, differs much from that which is derived from observation in corresponding circumstances of an invert sugar considered as a mixture of equal weights of glucose (dextrose) and levulose.

Clerget's numbers obtained in 1849† have been confirmed by Tuchschmid.‡ Tuchschmid's formula was

$$^{a}D = -(27^{\circ}.9 - 0^{\circ}.32t),$$

which is applicable to a liquor containing 17.21 gms. of inverted sugar in 100 cc. At different temperatures the rotatory power of pure levulose compared with the rotatory power of levulose calculated from invert sugar is as follows:

Temperature	0°	5°	10°	14°	20°
aD pure levulose . . .	101.22	98.42	95.62	93.38	90.02
aD levulose calculated from invert sugar . .	108.54	105.34	102.14	99.58	95.74

These differences are probably due to certain unexpected peculiarities produced by the action of acids upon the levulose.

(1). It is shown that the rotatory power of crystallized levulose is not affected by the introduction of equal weights of dextrose; this last sugar, therefore, cannot produce the difference noted.

(2). When pure levulose is subjected to the treatment by which invert sugar is made from cane sugar, it is found that its rotatory power is strongly affected. A solution of pure levulose which at 12° gave $^{a}D = -94^{\circ}.66$, gave $^{a}D = -96^{\circ}.78$ when it had been previously treated with five per cent. strong hydrochloric acid and heated to 68° for half an hour. When sulphuric acid was employed the rotatory power was still more highly increased, reaching $^{a}D = -99^{\circ}.77$. These results were not notably affected when the acids were carefully neutralized before the observation was made. Oxalic acid acts like the mineral acids in this particular. Acetic and formic acids are without effect. In 1846 Dubrunfaut§

*Comptes Rendus, Vol. 107, p. 390.
†Annales de Chimie et de Physique, 3d series, Vol. 26, p. 175.
‡Journal für prakt. Chemie, Vol. 2, 1870, p. 135.
§Comptes Rendus, Vol. 23, p. 38.

showed that the rotatory power of inverted sugar varied with the nature and proportion of the acid employed. The duration of the action of the acid is also not without influence. A solution of cane sugar inverted according to the method of Clerget showed the rotatory power of the levulose $[\alpha]_D = -101^\circ.3$; while the same solution inverted by another method, that is the addition of 5 per cent. of hydrochloric acid and heated to 68° for 30 minutes, showed $[\alpha]_D = -96^\circ.52$. It is also demonstrated that acetic acid and formic acids employed with 5 per cent. strength and at 100° invert cane sugar completely in 30 minutes and give a liquor whose rotatory power remains constant even after prolonged heating, and in the invert sugar thus obtained the levulose possesses the rotatory power of pure levulose. In the conditions where pure levulose gave $[\alpha]_D = -94^\circ.66$, we found after inversion with acetic acid for 30 minutes at 100°, $[\alpha]_D = -94^\circ.75$, and after heating for one hour, $[\alpha]_D = -94^\circ.48$; after heating with 10 per cent acetic acid for one hour, $[\alpha]_D = -94^\circ$, after heating with 5 per cent. formic acid at 100° for 30 minutes $[\alpha]_D = -94^\circ.80$. Moreover, it was seen that when hydrochloric acid was added to the solution inverted as above the levulose underwent modifications similar to those which pure levulose suffers in the same circumstances. The conclusions reached by these experiments were as following:

(1) Strong acids modify the rotatory power of levulose by increasing it more or less according to circumstances.

(2) The levulose of sugar inverted by the processes in ordinary use is not strictly identical with pure crystallized levulose.

H. W. W.

The Solubility of Sucrose in Distilled Water. In sugars approximately pure and dissolved in a constant volume, say 100 cc., and at a constant temperature, each gram increases by a constant volume even to the fourth decimal place for quantities ranging from 1 to 45 grams per 100, the densimetric expression of the solution; the rate of increase appears to be less regular from 45 per cent to [illegible] per cent, the maximum for solubility in the cold, and in supersaturated solutions because there is a sensible lowering of the primitive arithmetical value of the two consecutive terms from the value of 45 per cent, and that this new value, even to the

fourth decimal place, does not cease to grow slightly lower even up to 55 per cent. This constant increase from 1 to 45 per cent. is equal to $\frac{.0194}{5} = .00388$; from 45 to 50 it is equal to $\frac{.0186}{5} = .00372$; from 55 to 100 it is equal to $\frac{.0182}{5} = .00364$.

Notwithstanding these slight modifications, it is always easy to construct a table of density between 1 and 40 per cent. on one hand and from 55 per cent. to 100 per cent. on the other by taking in each series the density of the two terms which are near together. Each difference gives the coefficient sought for, being data of equal intervals.

Example.—Density of a 20 per cent. solution 1.0775, subtracted from the density of a 25 per cent. solution, viz., 1.0968 $=$ .0193 and $\frac{.0193}{5} = .00386$. In the second series the following example will suffice.

The density of a 60 per. cent. solution, 1.2289, less the density of a 55 per cent. solution, 1.2107, $= .0182$ and $\frac{.0182}{5} = .00364$.

The percentage of sucrose and the corresponding density are furnished approximately even to the third decimal place, and sometimes to the fourth decimal place, for values between 1 and 45 per cent. by the densimetric expression of a solution of 10 per cent. sugar reduced to its first four decimal places subtracted from unity divided by 10. Reciprocally, the division of the four decimal places of a density by the coefficient above reproduces practically the strength of the solution.

Example.—Density of a 10 per cent. solution, 1.0388 less unity $= .0388$; $\frac{.0388}{10} \times 45 = .1746$ plus unity $=$ the density of a solution containing 45 per cent. of sugar. The density 1.1746 minus unity $= .1746$; $.1746 \div \frac{.0388}{10} = 45$ per cent., the strength of the solution.

It is suggested that it may be possible in this way, having established exactly the coefficient for saccharose to have invert sugar or dextrose and determine the percentage of each of them in a solution by a simple comparison of the formula derived from its density.—(Perier, Comptes Rendus, Vol. 108, No. 23, p. 1202.)

H. W. W.

Detection of Dextrose by the Formation of Saccharic Acid. —The absence of a special reaction which can serve to detect dextrose or groups of dextrose led the authors to seek for a method as

sure as possible for the detection of that saccharine matter. The characteristic reaction of galactose in producing mucic acid on oxidation by nitric acid led to the expectation that the oxidation of dextrose by nitric acid might furnish some characteristic reaction, if that characteristic product, viz: saccharic acid, could be detected with absolute certainty. It was at first necessary to solve the question whether other glucoses might not furnish in the same conditions saccharic acid. To this end a series of experiments were instituted on the oxidation of the different glucoses by nitric acid. In order to avoid the presence in the product of oxidation of the nitric acid or of its salts, which peculiarly retard subsequent operations, the primitive operation of Sohst et Tollens* was modified, by evaporating the product to a sirupy consistence in place of evaporating it to one-third its volume. In some cases it was found that the nitric acid which remained in the product after this treatment could be entirely driven off by adding water and evaporating several times. Later, the quantity of nitric acid used was reduced in volume; in the greater number of experiments five or six parts of nitric acid at the density of 1.17 was employed for one part of the sugar. The hydrates of carbon which contain only a little of the dextrose, raffinose for example, might be treated with only four parts of the nitric acid. Sufficient elemination of the nitric acid is recognized by the fact that the sirup, up to this point colorless, commences to grow rapidly brown.

Oxidation of Dextrose.—5 grams of dextrose were evaporated to a sirupy consistence on a sand bath with 30 cc of nitric acid, with a density of 1.15. This syrup was redissolved in 20 cc. of water and the hot solution carefully neutralized by carbonate of potash. The solution by this process is colored brown. It is necessary to avoid a large excess of the alkali since it exercises a prejudicial influence; a few drops of acetic acid are then added, the liquid evaporated to a sirupy consistence and treated with a few drops of the same acid. By this treatment there is formed immediately bisaccharate of potash. The precipitate was placed upon a porous porcelain plate which absorbed the mother liquor. It was then redissolved in a small quantity of water and after recrystallization it was again placed upon the porcelain plate and

*Leibig's Annalen, Vol. 245, p. 1.

washed with a small quantity of water by means of a spray bottle. This operation was repeated until the saccharate became free from oxalic acid which is always formed during oxidation. In order to establish the identity of the saccharic acid its silver salt was prepared. To this end the bisaccharate of potash was dissolved in water, filtered, neutralized by ammonia and a solution of the nitrate of silver added to it the content of which in nitrate of silver represented 1.5 times the weight of the bisaccharate of potash. The neutral saccharate of silver formed a milky precipitate becoming pulverulent if the liquid was stirred a long time. This precipitate was filtered, washed with a small quantity of water and dried in a dark place over sulphuric acid. 5 grams of dextrose furnished 3.6 gms of crude bisaccharate of potash, 1.8 gms of the pure salt and 2 gms of the saccharate of silver. Upon analysis the salt furnished 50.81 per cent. of silver, theory, 50.94 per cent. ($C_6H_8Ag_2O_8$).

Cane Sugar.—Cane sugar treated as above and in the same quantity furnished only 1.1 gms of saccharate of silver. The analysis of the dried salt gave 50.44 per cent. silver, theory 50.94 per cent.

Inuline.—In the oxidation of inuline as above, neither mucic nor saccharic acid was obtained.

Galactose.—Three sets of experiments were made with galactose employing each time 5 gms. of substance. In two cases this quantity of galactose was oxidized by 30 cc of nitric acid, and in the third case by 20 cc. The galactose gave rise in its oxidation to a large quantity of mucic acid which so thickened the product of the reaction that it was difficult to drive the nitric acid off by a single evaporation. In our first experiment the galactose furnished 76.94 per cent. of mucic acid ; the filtered portion neutralized by the carbonate of potash and treated with acetic acid gave in addition a notable quantity of nitrate of potash, in such a way that it was impossible to detect the presence of saccharic acid.

In the two experiments there was added after evaporation a small quantity of water, the substance reevaporated and this operation repeated until the nitric acid seemed to be eliminated. The yield in mucic acid was 79.16 per cent.; the filtered portion was again treated with carbonate of potash, evaporated and treated with acetic

acid. There was found a crystalline deposit which was purified by exposure on a porous porcelain plate and repeated crystallization. This deposit contained in addition to some needles of nitrate of silver a crystalline powder; this powder slightly soluble in cold water, very soluble in hot water, contained still some oxalic acid and was too small in quantity to be subjected to analysis. In the third oxidation with 20 cc. of nitric acid the product contained still after the first evaporation some nitric acid, which was driven off after four successive evaporations. The yield in mucic acid in this case was 63.6 per cent. The filtered portion after concentration was treated with carbonate of potash and acetic acid, and furnished a deposit like that described above. Freed from oxalic acid this deposit weighed .4 gm. In order to transform the potassic into the silver salt, nitrate of silver was added, and this formed immediately a precipitate which resembled saccharate of silver, but its small quantity rendered the determination of silver therein impracticable. The attempt therefore to prepare separately the saccharate of silver from 5 gms. of galactose was not successful, since the small quantity obtained of the silver salt could have come from the mucic acid remaining in solution.

Sorbose.—The oxidation of 5 gms. of sorbose by 30 cc. of nitric acid furnished neither mucic nor saccharic acid.

Arabinose.—5 gms. of arabinose were oxidized by 20 cc. of nitric acid; after the first evaporation the product did not contain nitric acid. No mucic acid was formed. The product of oxidation was neutralized by carbonate of potash, evaporated, and treated several times by acetic acid; it deposited a crystalline body which under the microscope was found to be composed of small triangular crystals. After two re-crystallizations this precipitate weighed .46 gm. By the addition of silver nitrate the solution of this body became readily colored and produced a black precipitate which contained some silver. The yield was too small to be submitted to analysis. From the aspect and other properties of this precipitate, it could not have been the neutral saccharate of silver.

Lactose.—Two experiments were made with milk sugar; in the one 30 cc. of nitric acid were employed and in the other 20 cc. In the first experiment 36.8 per cent. of mucic acid was obtained;

the filtered portion was treated as above described for the saccharate of potash. 1.36 gm. of the crude bisaccharate of potash was obtained, .65 gm. of pure bisaccharate and .7 gm. pure neutral saccharate of silver. The salt contained of silver 50.35 per cent. theory 50.94. In the second experiment with 20 cc. of nitric acid at the end of the first evaporation the whole of the nitric acid was used up. We obtained in this a marked deficit of mucic acid, viz; only 25.4 per cent.; nevertheless an increase in the saccharic acid, viz; 1.55 gm. of the crude and .08 gm. of the pure saccharate of potash and .75 gm. saccharate of silver. The percentage of silver obtained was 50.43, theory 50.94.

Raffinose.—Three experiments were undertaken with raffinose with 5 gms. each of the substance; the first two with 30 cc. of nitric acid and the third with 20 cc. Since raffinose behaves exactly like lactose the method employed with that was pursued. The first experiment in which the oxidation was more rapid than by the second gave 18.6 per cent. mucic acid, the second 19.32 per cent. The first experiment afforded .95 gm. crude saccharate of potash, pure .44 gm., saccharate of silver .43 gm., containing of silver 50.82 per cent. In the second experiment there was obtained 1.05 gm. of the crude and .65 gm. of the pure acid saccharate of potash and .65 gm. of the neutral saccharate of silver, containing 50.29 per cent. In the three experiments at the end of the first evaporation the 25 cc. of nitric acid were completely used up. The experiment afforded only 11.86 per cent. of mucic acid; nevertheless a considerable amount of saccharic acid, viz: 1.25 gm. crude, .70 gm. pure acid saccharate of potash, and .70 neutral saccharate of silver containing 50.27 per cent. of silver. From each 5 gms. of the hydrates of carbon, which, according to previous research, gave by hydrolysis dextrose, viz.: starch, lactose and sucrose, a potash salt and silver salt of the composition and property of saccharic salts were obtained. In the same manner this salt was obtained from raffinose in which the presence of dextrose was suspected. From the other hydrates of carbon which were examined no saccharic acid was obtained. According to our expectations, therefore, it has been established that by treatment of 5 gms. of any carbohydrate as above described, a certain reaction for dextrose is obtained.

A MORE GENERAL EXAMINATION OF THE SACCHARIC ACID ARISING FROM RAFFINOSE.

In order to obtain pure crystallizable saccharic acid from lactose and raffinose the following method was employed:

150 gms. of raffinose were oxidized with 750 cc. of nitric acid and 200 gms. of lactose with 1000 cc. of nitric acid, specific gravity 1.15. The 150 gms. of raffinose yielded 31 gms. mucic acid = 20.7 per cent. and 11 gms. of pure acid saccharate of potash. The 200 gms. of lactose yielded 70.5 gms. of mucic acid = 35.25 per cent. and 24.25 pure acid saccharate of potash. The neutral saccharate of silver was prepared by adding the saccharic acid solution to the silver nitrate solution. The free saccharic acid prepared from a silver salt by means of hydrochloric acid was digested with a little bone-black, filtered and evaporated to a thick sirup, after the addition of a few crystals of saccharic acid the sirup crystallized to a hydroscopic mass which had no certain melting point, varying from 90° to 100°, and which did not show any four-sided crystals under the microscope. This mass did not dissolve completely in water, and the part insoluble in water was proved to be mucic acid by its melting point of 210°. The sirup again evaporated, not yet entirely free from mucic acid, was dissolved in absolute alcohol, in which mucic acid is insoluble, and after filtration again evaporated to a sirupy consistence. The part insoluble in alcohol melted at 210°, and was, therefore, mucic acid. The sirup crystallized after several days in crystals with a silk-like lustre, which were dried upon porous porcelain. After the second crystallization the crystals melted at 124° to 127°; they were weighed, but contained still a trace of ash. Dried over sulphuric acid, they gave the following numbers on analysis:

	1	2	Theory: $C_6H_8O_7$
Carbon	36.95	37.39	37.50
Hydrogen	4.22	4.26	4.17

The acid obtained from raffinose possessed the composition of the saccharo lactonic acid from starch. The crystals dried over sulphuric acid did not reduce Fehling solution, but when dried at 100° became strongly reducing. The melting point 124° to 127° was not so high as that observed by Sohst and Tollens, viz.: 130°. It is believed, therefore, on account of the properties of the acid

obtained, that it contains saccharic acid, arising from the presence of dextrose in raffinose. Raffinose is, therefore, composed of galactose, levulose and dextrose. (Gans & Tollens, Liebig's Annalen, Vol. 249, Nos. 2 and 3, p. 215.) H. W. W.

Butter Analysis.—L. F. Wilson, Ztschr. anal. Chem. **28,** 175. The clear ethereal solution of butter fat obtained from the milk by extraction, according to the method of Soxhlet (see this Journal **1,** 124), is poured into a small beaker and the ether driven off by a gentle heat and the fat dried in an air bath at 100° C. for half an hour.

Of the still warm fat 2.5 gms. are poured down a glass rod into a 200 cc. flask, allowed to solidify, and then any excess removed, so that what is left shall weigh exactly 2.5 gms.

The fat is then saponified with 5 cc. of alcoholic potash solution, made by adding 20 gms. pure caustic potash to 100 cc. 70 per cent. alcohol. Saponification is accompanied by an odor resembling that of butyric ether*, and is completed in a few minutes if the flask is heated over a small flame. If the flask is shaken a few minutes longer most of the alcohol is driven off. The remaining alcohol, which is expelled only with difficulty, may be driven off by placing the flask in boiling water and drawing air through it by means of a filter pump. The heating and passing of air is continued until the soap no longer swells up in bubbles when the air in the flask is rarified (the soap from oleomargarine swells up under these conditions far less than the butter-fat soap). It is very important to drive off the last trace of alcohol, as otherwise ethers may be formed in a subsequent stage of the process and part of the volatile acid be combined and overlooked.

The soap is then dissolved in the flask in 50 cc. water, some pieces of pumice stone (to prevent bumping) and 20 cc. of orthophosphoric acid (sp. gr. 1.125) added. The flask is connected with the condenser by a wide tube inclined upwards and upon which a glass bulb is blown to catch and return to the flask any particles of solution carried up mechanically. The flame is so regulated that 1 cc. of distillate per minute is obtained. 50 cc. are distilled and filtered through a wet filter to free it from insoluble fatty acid which also distills over in small quantity. The

*Oleomargarine gives no such odor.

filtrate is made up to 100 cc., a couple of drops of dilute phenol-phtalein solution added, and titrated with tenth normal caustic potash solution as free from carbonate as possible.

The author has also made the following observations concerning the amount of volatile acids in butter fat.

1. The colostrum fat is very poor in volatile acids, in two cases only 9.27 and 10 cc. alkali were needed.

2. The volatile acids increased very rapidly for from 5–7 days after calving, and then slowly decreased until the end of the lactation period is reached. It seldom falls below 12 cc. of alkali, and is not affected either by the season of the year or by the food.

3. The volatile acids vary decidedly in different cows.

4. The amount of alkali used varies in normal milk from single cows between 11.45 cc. to 20.5 cc.

5. If the cow is in heat, or sick, the alkali used sometimes falls below this minimum.

A Modification of the Kjeldahl Method. J. W. Gunning. Ztschr. anal. Chem. **28**, 189. 1 part potassium sulphate is mixed with 2 parts ordinary sulphuric acid and heated. The resulting mass contains $KHSO_4$, and is at ordinary temperatures half solid. It melts easily, and may be easily poured from warm vessels. 500–1000 milligrams of the substance to be examined is placed in a flask containing [illegible] with 20–30 cc. of the acid mixture and heated. Liquids should be first treated with sulphuric acid in the flask and evaporated nearly to dryness.

At first the substance is apt to froth considerably, accompanied by a loss of acid. This loss of acid should not be allowed to go too far, as the temperature then rises and there is danger of a loss of ammonia. To prevent this the flame should be so regulated that the acid condenses on the sides of the flask and flows back. The time required for complete decomposition is from [illegible] to 2 hours. To determine the ammonia the author prefers sulphuric acid as an absorbent. The excess of acid is best determined by adding sufficient potassium iodate and iodide and determining the iodine with sodium thiosulphate.

Short's Method of Fat Estimation in Milk. Kreitmair. Zeitschrift für angewandte Chemie, [illegible], p. 288.

Comparative determinations are given for Soxhlet's areometric and Short's method. In most cases the quantity of fat as determined by Short's method was greater than by the ether process. The milk was bought in open market. Some of the numbers obtained are given below:

(*Samples in which Short's method gave lower results.*)

No.	*Fat by Soxhlet.*	*Fat by Short.*
2	2.07	2.05
3	2.24	2.15
4	2.84	2.39
5	3.00	2.69
6	2.19	2.14
7	2.30	2.24

Samples in which Short's method gave higher numbers.

No.	*Fat by Soxhlet.*	*Fat by Short.*
8	2.97	3.09
9	3.04	3.18
10	3.60	4.29
11	2.84	2.95
12	3.33	3.89
13	2.99	3.69
14	3.76	4.48
15	3.76	4.19
16	3.98	4.58
17	1.96	2.39
18	4.46	4.98 4.98 4.98
19	1.65	1.70 1.65 1.60

Triplicates by Short's method agree well together. The cause of the discrepancies in the two sets of operations was not discovered.

H. W. W.

The Free Fatty Acids in Oils.—H. Noerdlinger, Ztschr. anal. Chem. **28,** 183. The oil is dissolved in ether and alcohol, phenol-phtalein added and titrated with tenth normal caustic potash. 3 varieties of oil were examined by the author.

1. Pressed oils suitable for food.
2. Pressed oil for technical use.
3. Oil extracted by means of light petroleum.

The first and second sorts were pure commercial articles, the third sort was prepared in the laboratory from the pulverized fruits.

a. FLUID OILS.

	Fatty Acids.		
	Minimum.	*Maximum.*	*Mean.*
1. Rubsen Seed Oil.—			
Salkowski found in commercial oil,	—	—	4.28
V. Rechenberg " " "	—	—	6.64
3 sorts pressed food oil	0.53	1.82	1.19
9 " technical oil	0.52	6.26	2.88
2 " extracted oil	0.77	1.10	0.93
2. Olive Oil.—			
Salkowski found in commercial oil,	—	—	1.17
V. Rechenberg " " "	—	—	2.25
1 sort food oil	—	—	1.66
3 sorts technical oil, purity questionable	3.87	27.16	12.97
3. Poppy Oil.—			
Salkowski found in commercial oil,	—	—	2.29
V. Rechenberg " " "	—	—	2.09
26 sorts pressed food oil	0.70	2.86	1.92
5 " technical oil	12.87	17.73	15.37
5 " extracted oil	2.15	9.43	4.72
4. Ground Nut Oil.—			
Salkowski found in commercial oil,	—	—	1.66
13 sorts pressed food oil	0.85	3.91	1.94
12 " technical oil	3.58	10.61	6.52
16 " extracted oil	0.95	8.85	4.02
5. Sesame Oil.—			
14 sorts pressed food oil	0.47	5.75	1.97
7 " " technical oil . . .	7.17	33.13	17.94
7 " " extracted oil . . .	2.62	9.71	4.89
6. Cotton-Seed Oil—			
Salkowski found in commercial oil,	—	—	0.29
1 sort pressed food oil	—	—	0.15
2 sorts " technical oil	0.42	0.50	0.46
7. Oil of Mustard.—			
2 sorts pressed oil	0.68	1.02	0.85

	Fatty Acids. Minimum.	Maximum.	Mean.
8. Castor Oil.—			
9 sorts pressed oil	0.62	18.61	9.28
5 " extracted oil	1.18	5.52	2.78
9. Linseed Oil.—			
Salkowski found in commercial oil,	—	—	3.45
V. Rechenberg " " "	—	—	1.74
10 sorts pressed technical oil	0.41	4.19	1.57
10. Candle-Nut Oil.—			
The candle nuts contain 61 per cent. of a thick fluid oil, from which solid glycerides separate at the ordinary temperature.			
1 sort pressed technical oil (about 3 years old)	—	—	56.45
b. SOLID FATS.			
11. Palm Kernel Oil.—			
Salkowski found in 2 sorts of commercial oil	13.26	13.39	13.33
27 sorts pressed technical oil	3.30	17.65	6.91
10 " extracted oil	4.17	11.42	8.49
12. Palm Oil.—			
1 sort (preparation unknown)	—	—	50.82
13. Cocoanut Oil.—			
Salkowski found in commercial oil,	—	—	2.96
9 sorts pressed technical oil	3.03	14.35	7.92
3 " extracted oil	1.00	6.31	4.26
14. Ilippe Fat.—			
Pressed technical oil (3 years old)	—	—	28.54
15. Niam Fat.—			
2 sorts extracted oil	14.40	34.72	24.56
16.—Bicuhyba Fat (myristica bicuhyba).—			
1 sort pressed oil (3 years old)	—	—	18.55
17.—Japan Wax.—			
Commercial wax (preparation unknown)	—	—	9.25

The small amount of free acid in cotton seed oil is due to the caustic soda used in preparing it for market. In natural oils as above shown the amount varies within wide limits, due no doubt to the difference in locality in which the seed was grown, temperature, moisture, etc. The author points out that food oil should be low in free acid.

Identification of Sulphonal.—Wefers Bertink. Niew Tydschrift voor Pharmacie 1889, through Pharm. Rund. If a few crystals of sulphonal are mixed with powdered iron and heated in a test tube, vapors with the odor of garlic are given off and sulphide of iron is formed. This is examined by adding acid, when sulphuretted hydrogen is given off, which blackens paper moistened with sugar of lead solution. The iron used must of course be first tested for sulphur in a blank experiment.

Identification of Cocaine. Greitherr. Pharm. Rund. from Pharm. Zeit. 1889, 617. If a few drops of a cocaine solution are mixed with 2–3 cc. chlorine water and 2–3 drops of palladious chloride solution, containing 5 per cent. salt, are added, a beautiful red precipitate is formed, which is slowly decomposed by water, is insoluble in alcohol and ether, but soluble in sodium thiosulphate. Other alkaloids do not show this reaction.

Some Results of Analyses of Olive Oil from Various Sources. L. Archbutt. J. Soc. Chem. Ind. **8**, [illegible]

Oil from the Seed of Jatropha Curcas. F. M. Horn. Ztschr. anal. Chem. **27**, [illegible]. This oil which was formerly used in medicine under the names Oleum ricini majoris and Oleum infernale, is pressed from the seed of Jatropha curcas. It is now being imported into Germany from Portugal in considerable quantities for burning and making soap. It may also be used as an adulterant for olive oil and for the preparation of turkey red oil. It has a very [illegible] characteristic odor and slight yellow color. At [illegible] white flakes of vegetable stearin begin to separate and at [illegible] is quite solid. The [illegible] soluble in alcohol [illegible] this way [illegible] castor oil which [illegible] in chloroform [illegible] saponification [illegible] potash as might be expected [illegible] in alcohol. The test was

to effect complete saponification is to heat the oil in contact with the finely divided caustic potash on the water bath for 10 minutes, and then add alcohol. The caustic potash was obtained by evaporating a known volume of a standardized caustic potash solution. The following constants for the oil were determined: Density at $15°$ C.=0.9192; Hehner's number=87.9; Reichert's number=0.65; Köttstorfer's number=230.5; Hubl's number=127.

Determination of Acetone.—E. Hintz, Ztschr. anal. Chem. **27**, 182. In 1880 G. Krämer described* a method for this purpose which is still in use. It consists in adding 1 cc. of the methyl alcohol to 10 cc. twice normal soda solution in a 50 cc. stoppered cylinder, then adding 5 cc. of a double normal iodine solution and shaking. In this way, as Lieben had before† pointed out, the acetone present is converted into iodoform. The iodoform is then dissolved out by 10 cc. of ether, and 5 cc. of the ether evaporated on a watch glass and the residue weighed. This residue multiplied by 0.28 gave the amount of acetone in 1 cc. alcohol. If very small amounts of acetone are present the results obtained are too high, due to a little potassium iodide dissolved in the ether. This error may be corrected by volatilizing the iodoform and weighing the residue. If much acetone is present, the results are too low, and in such cases it is necessary to decrease the amount of methyl alcohol taken, so that the iodine shall be present in greater excess.

Heintz, however, finds that it is best to make a blank analysis with reagents used, and subtract the iodide obtained in this way rather than to volatilize the iodoform. It is also necessary to dilute methyl alcohol rich in acetone with water, so that the acetone present shall not exceed 1–1.5 per cent.

It sometimes happens that crude methyl alcohol contains tarry impurities, which are separated and determined with the iodoform. In such cases it is necessary to make a blank experiment, leaving out the iodine.

The Testing of Wines for Nitric Acid.—According to Egger‡ the addition of water to wine may be detected by testing for ni-

*Ber. **13**, 1000.
†Ann. Chem. Pharm. Suppl. **7**, 218, 377.
‡Ztschr. Anal. Chem. **24**, 620.

trates, which are contained in all ordinary well water and are not contained in natural wines. If a white wine, 100 cc. is evaporated to a thin syrup, precipitated with absolute alcohol, the filtrate evaporated with the addition of water and animal charcoal to 10 cc., giving a clear solution for testing. Red wines are first precipitated with sugar of lead, filtered hot, the excess of lead removed by means of magnesium sulphate, and the process finished as with white wines. The clear white solution obtained in either case is tested for nitrates by pouring several drops into a dish in which is placed some crystals of diphenylamine and 1 cc. concentrated sulphuric acid. The production of a blue color shows presence of nitrates.

Borgmann has examined this method and notes the following:

(1). In a few instances natural wines contain nitrates.

(2). Wines to which nitrates have been added, sometimes, after standing, no longer give the reaction. This is probably because the ferments present have destroyed nitric acid.

(3). Since water containing nitrates is used to wash the casks, in some cases, where water used is very rich in nitrate, the wine may contain nitrates and be a perfectly natural product.

Borgmann finds that by the process described by Egger, .05 milligramme of nitric acid per liter may be detected. The test is most delicate with a solution made by dissolving .01 gm. diphenylamine in 100 cc. concentrated sulphuric acid. The animal charcoal used should be carefully tested for nitrates by heating it with water and testing the filtrate.

Rapid Determination of Sugar with Fehling's Solution.—J. E. Politis, Chem. Zeit. Rep. **13**, 221. For a known amount of sugar solution an excess of $\frac{n}{10}$ copper solution is used and after the reduction the excess of copper is titrated, according to Haen's method, with potassium iodide and sodium thiosulphate.

The copper solution contains 24.95 gms. crystallized copper sulphate, 140 gms. potassium sodium tartrate, and 25 gms. caustic soda to the liter. The $\frac{n}{10}$ thiosulphate solution contains 24.8 gms. to the liter and is standardized with $\frac{n}{10}$ iodine solution containing 12.7 gms. iodine to the liter. 1 cc. of the copper solution will reduce 0.0036 gm. of glucose.

50 cc. of the copper solution is heated to boiling in a porcelain

dish and 10 cc. of the sugar solution (which should contain about one part to the thousand of glucose) is added and boiled five minutes.

The solution is then diluted to 100 cc. and exactly 50 cc. taken for analysis. This is slightly acidified and a small excess of potassium iodide and starch solution are added. The iodine set free by the excess of the copper solution is titrated with the thiosulphate solution and the amount of the copper solution reduced by the sugar is easily calculated. S. C.

Determination of Formic Aldehyde.—Legler has shown* that either of the following reactions may be made use of in the determination of formic aldehyde:

$$(1).\ 6CH_2O+4NH_3=(CH_2)_6N_4+6H_2O.$$
$$(2).\ 2CH_2O+NaOH=HCO_2Na+CH_3OH.$$

Of these he preferred the first. The ammonia was added in slight excess, and the excess determined with dilute standardized sulphuric acid.

G. Lösekann finds† that the hexamethylene-amine produced, neutralized a part of the acid, and must be taken into consideration. It behaves as a monacid base, and in the titration $4CH_2O$ is therefore equivalent to $1H_2SO_4$. Either methyl orange or cochineal may be used as indicator. If methyl orange is used the sulphuric acid should be added until a full red color is produced, as this marks complete neutrality.

Eschweiler‡ says that Lösekann is correct if the indicator be methyl orange or cochineal, but that if litums or phenol-phtalein be used, the method of calculation employed by Legler must be used. In other words if litums or phenol-phtalein is used, for every 6 molecules of formic aldehyde 4 of ammonia will be indicated, while with methyl orange and cochineal only 3 molecules will be shown. Congo red and tropaeoline behave like methyl orange.

Eschweiler finds also that with dilute solutions it is necessary to allow the mixture of formic aldehyde and ammonia to stand for a day or so to complete the reaction, or to heat it for a short time to 100°.

*Ber. **16**, 1333. †Ber. **22**, 1565. ‡Ber. **22**, 1929.

Analysis of Ethereal Oils.—Benedikt and Grüssner, Chem, Zeit. **13**, 1087. The authors use Teisel's method, in which the oil is heated with hydriodic acid, and the iodide of the alcohol radical formed is decomposed with silver nitrate. The silver iodide thus obtained is weighed, and the equivalent weight of the alcohol radical, calculated for one gram of oil, is the "methyl number."

For example, if 0.2963 gm. oil gives 0.3737 gm. silver iodide, which is equivalent to 0.02385 gm. methyl (CH_3), then 1 gm. oil is equivalent to 0.0805 gm. methyl, and the "methyl number" would be 80.5.

Oils to be tested must be free from alcohol; but if the "methyl number" of the oil is known, the percentage of alcohol may be determined. The "methyl number" of absolute alcohol is 326. Oils containing sulphur give a black precipitate with the silver solution, and produce inaccurate results. Oils containing the radicals of higher alcohols do not leave a clear solution over the silver iodide precipitate, even after repeated heating with hydriodic acid.

The following table gives the methyl numbers determined by the authors. Those marked zero gave only a trace of silver iodide.

No.	No. in Kremel's Tables.	Name. (In Latin)	"Methyl Number."
1	1	Ol. Absinthii	0
2	2	" Amygdlar. amar.	0
3	6	" " "	0
4	10	" Angelicae	0
5	12	" Anisi	82.8
6	18	" " stellati	79.8
7	22	" Aurant. florum	0
8	21	" " corticum	6.9
9	26	" Bergamottae	0
10	28	" "	6.6
11	—	" Betulini	22.4
12	35	" Calami	24.2
13	45	" Carvi	0
14	46	" "	8.3
15	47	" "	0
16	42	" "	6.6
17	36	" Caryophyllorum	88.8
18	39	" "	86.9
19	40	" "	73.8

No.	No. in Kremel's Tables.	Name. (In Latin.)	"Methyl Number."
20	50	" Cassiae florum	6.7
21	51	" " foliorum	73.2
22	55	" Cinnamomi	25.7
23	59	" Citri cort	0
24	58	" " "	23.6
25	61	" Copaivae	0
26	63	" Coriandre	0
27	64	" Cubebarum	0
28	65	" Elemi	12.1
29	67	" Eucalypti	0
30	69	" Foeniculi	65.7
31	73	" Gaultheriae artefact	89.4
32	79	" Geranii	0
33	77	" "	0
34	76	" "	0
35	80	" Juniperi	0
36	81	" "	0
37	86	" Laurocerasi	13.8
38	85	" "	0
39	93	" Lavandulae	0
40	87	" "	2.4
41	97	" Menthae Crisp.	0
42	101	" " pip.	0
43	107	" " "	0
44	105	" " "	0
45	109	" Olibani	9.1
46	110	" Petroselini	92.2
47	111	" Pini pumil	0
48	116	" Pulegii	0
49	129	" Rosmarini	0
50	124	" Sabinae	0
51	125	" Salviae	0
52	126	" Santali	0
53	128	" "	0
54	138	" Terebinthinae	0
55	136	" "	0
56	137	" "	0
57	141	" Valerianae	0

When a name is repeated it indicates a different brand of oil.

The value of some oils may be determined from the "methyl number." The per cent. of eugenol, $C_6H_3(C_3H_5)(OCH_3)OH$, in No. 17, whose "methyl number" or percentage of methyl is

88.8, is found to be 97.0, while the next brand whose "methyl number" is 86.9 gives 94.0 per cent.

[NOTE.—Evidently there is a mistake in the decimal points of the above "methyl numbers." To obtain the results given they should *all* be removed one figure to the left, i. e., 88.8 should read 8.88. S. C.]

NOTES.

—Dr. A. R. Palmer has been elected Professor of Chemistry in the University of Illinois at Champaign.

—Eikonogen (sodium amido-*Beta*-naphtol-*Beta*-sulphonate, $C_{10}H_5NH_2OH. SO_3Na$) is proposed by Dr. Andreson of Berlin as a new photographic developer. It is manufactured by the Actiengesellschaft für Anilinfabrikation, and according to Andreson gives minute detail, a tone better than pyrogallol, keeps well, is not poisonous, and does not cause the reduction of unaltered silver chloride. (Chem. News **60**, 103.)

—*The Proposed American Chemical Society.*—In undertaking a new enterprise it is well to first carefully count the cost, and having done so, to proceed vigorously and fearlessly. Are the conditions such that an American Chemical Society could be successful? We think it will be evident to any one who has given the matter careful study, that a membership of 500 is all that could be expected for several years, and it is also evident that the annual fee should be fixed at not more than $5. This would make an income not exceeding $2500. If a publication is contemplated, and the pay of the editor and his office expenses, etc., are deducted, very little would be left for the journal. It is possible that the financial question might be successfully met; but those who have had experience in running journals are not apt to be sanguine upon the finance problem. Then as to the place of meeting. It has been proposed to organize the Society in affiliation with the American Association for the Advancement of Science, the meetings to be held at the same time and place. It is clear that this can only be done in one of two ways. 1st, by making membership in the Association a condition of membership in the new Society. If this were done, the addition of $3 paid to the Association, making a total fee of $8, would deter a

great many persons from joining. It seems to us very doubtful whether this plan would result in a very large membership. 2d, the new Society would hold its meetings at the same place and time, but have no official connection with the Association; but this plan is plainly unfair to the Association, for by it the Society takes advantage of the arrangements made for the Association, and renders no equivalent in return.

At the Toronto meeting of the American Association, a committee was appointed by the chemical section to consult with committees of other societies as to the advisability of forming an American Chemical Society; but the motion to appoint this committee was only carried by a majority of 3 at a thinly attended session and while part of the members were away upon an excursion and another part had gone home, having previously been negatived. When the Council of the Association came to pass upon this proposition, it declined unanimously to have anything to do with it. For these reasons it seems to us evident that such a Society affiliated with the American Association could not be successful.

In organizing a separate society, the difficulties to be faced are the financial one and the arrangements for meeting. Whether these can be surmounted, can only be determined by trial. The New York Chemists have tried to form an American Chemical Society and have failed. The chief support of the new movement seems to come from the Washington chemists. If they wish to undertake it anew, let them do so by publishing the main lines upon which the proposed society is to be constructed, and then get signatures from those willing to join. This is the only rational way to go about such things. Further discussion of the matter seems to us to be only likely to make more pronounced the already marked difference of opinion, and to develop an opposition that would certainly be fatal. E. H.

—The Laboratory of the Crane Iron Company, at Catasauqua, Pa., was burned on September 25, at 2.15 A. M. The platinum ware and balances were saved.

—Crampton has found boric acid in 34 samples of wine. According to the same authority, boric acid is contained also in the ash of the peach and watermelon (Ber. **22**, 1072).

—According to George Kassner (Chem. Zeit. **13**, 1302), pure oxygen may be rapidly obtained by adding caustic potash solution to a mixture of potassium ferricyanide and hydrogen dioxide

$$K_6Fe_2(CN)_{12} + 2KOH = K_8Fe_2(CN)_{12} + H_2O + O$$
$$H_2O_2 + O = O_2 + H_2O$$

58 gms. potassium ferricyanide with 100 cc. 3 per cent. hydrogen dioxide gives 2 liters of pure oxygen. S. C.

OBITUARY.

—Professor Victor Eggertz died in Stockholm, the last week in August, at the age of 72. He was for several years at the head of the School of Mines in Sweden, first at Fahlun and then at Stockholm. His invention of the so-called coloration test for carbon analysis in iron and steel has been of great practical value (Chem. News **60**, 119.) A. H. W.

NEW BOOKS.

Brown's Assaying.* The three editions of this work issued successively in 1883, 1886, and 1889, show the popular estimate placed upon the book. The author states in his "Notes to the Third Edition" that it differs from the previous one only in the correction of a few typographical errors, in the revision and correction of the assayers' outfit, and some minor alterations in the descriptions of apparatus. At least one fourth of the second edition, we learn also, has been sold in England, Australia and Germany. This is as it should be. If our scientific men write good books, they will surely find a market abroad. E. H.

Tillman's Lessons in Heat.† This book, prepared to meet the necessities of a short course of study in the subject of heat, is admirably suited to the purpose. It gives the salient points of the science without confusing the student with a multiplicity of experiments. The experiments are well selected and the descriptions crisp and correct.

*[illegible] With [illegible] By Walter Lee Brown, B. Sc. Third Edition [illegible] H. Sargent [illegible]

†[illegible] Heat. By S. E. Tillman, Professor of Chemistry [illegible]

Several slight defects in statement appear: the one which causes so much trouble that "warm air rises" being conspicuous. Is it not time to give "lumineferous ether"—*the ether*—the prominence it deserves? The evidence in favor of its existence is as strong as that of any other medium, and no detriment would come to scientific truth and accuracy, or to the student to speak of it as if it did exist.

Ether waves would, naturally, take their place under radiation, absorption, etc., and the artificial distinction between light and radiant heat disappear, much to the advantage of science. It is a pity that more time cannot be given to the study of the subject of heat by the cadets at West Point.

The publishers have presented a book which is not repugnant in appearance. The paper, typography and illustrations are good. J. W. M.

Mason's Notes on Qualitative Analysis.*—This little book contains the usual tests for the bases and acids, followed by the scheme for complete analysis. As special features, the author has inserted a number of experiments under the tests for nearly every base and acid, and also given questions which must be answered by consulting works of reference. This idea is an excellent one, and, if carried out, will make a decided improvement in the subsequent work of the student. The book has an appendix which gives the preparation of reagents, colored flames, examination on charcoal, and the care of platinum vessels. S. C.

Bernthsen's Organic Chemistry.†—The English edition of this work has been carefully revised and brought up to date by the author and translator. The historical part is omitted, but the names of the original workers are given with the compounds, together with the date of the work. References are made to the original papers in all the important compounds. Parts of minor importance are printed in smaller type.

*Notes on Qualitative Analysis, arranged for the use of students of the Rensselaer Polytechnic Institute, by W. P. Mason, Professor of Analytical Chemistry. Second edition. 44 pp., cloth. Nims & Knight, Troy, New York.

†A Text-Book of Organic Chemistry. By A. Bernthsen, Ph. D., formerly Professor of Chemistry in the University of Heidelberg. Translated by George M'Gowan, Ph. D., Demonstrator in Chemistry, University College of N. Wales, Bangor.

The original text specially brought up to date by the author for this edition. 12 mo., 544 pp. D. Van Nostrand Co., 23 Murray St., New York. 1889.

The work is condensed as much as possible, and yet the descriptions are perfectly clear, so that the student will have no trouble in understanding them. This is a very desirable feature in a text-book. The introduction gives the methods of analysis of organic compounds, calculation of formulæ, and chemical theories. The remainder of the work treats of the preparation and properties of the paraffine series, carbohydrates, and benzene derivatives. The paper and print are good. S. C.

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